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REACTOR FUEL PROCESSING

A Quarterly Technical Progress Review

Prepared for DIVISION OF TECHNICAL INFORMATION, USAEC, by
STEPHEN LAWROSKI and ASSOCIATES, ARGONNE NATIONAL LABORATORY

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REACTOR FUEL PROCESSING

A REVIEW OF RECENT DEVELOPMENTS

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Foreword

This quarterly review of reactor fuel processing has been prepared at the request of the U. S. Atomic Energy Commission, Division of Technical Information. It is intended to assist those interested in keeping abreast of important developments in this field. In each Review it is planned to cover those particular subjects in which significant new results have been obtained. The Review does not purport to abstract all the literature published on this field during the quarter. Instead it is intended to bring each subject up to date from time to time as circumstances warrant.

Interpretation of results, where given, represents the opinion of the editors of the Review, who are personnel of the Argonne National Laboratory, Chemical Engineering and Idaho Divisions. Those taking part in the preparation of this issue are J. Barghusen, L. Burris, Jr., P. Fineman, A. A. Jonke, S. Lawroski, W. J. Mecham, D. Paige, J. Royal, W. B. Seefeldt, J. Simmons, R. K. Steunenberg, C. E. Stevenson, and R. C. Vogel. The reader is urged to consult the original references for more complete information on the subject reported and for the interpretation of results by the original authors.

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Section

I

Reactor Fuel Processing

Commercial Aspects of Fuel Processing

Fuel-Processing Plans

Domestic Plans

After completing public hearings in April, the Atomic Energy Commission (AEC) announced in May 1963 that a permit had been issued to Nuclear Fuel Services, Inc. (NFS), and the New York State Atomic Research and Development Authority for construction of a plant to process irradiated fuel.[1] A ground-breaking ceremony was held on June 13. The latest date for expected completion of construction is Dec. 31, 1965.

The AEC has agreed in principle to provide NFS with a work load equivalent to about 625 days of operating time over a five-year period beginning in 1966. This commitment might be slightly increased, at the AEC's option, or decreased if the commercial fuel load proves to be much heavier than expected.[2]

The AEC has prepared the analysis shown in Table I-1 for the types of fuel it expects to include in the 625-day base load, together with the costs involved.

The AEC has also estimated that the chemical-processing work load for all nongovernment reactors up to the year 1980 will be as follows in terms of the NFS plant's capacity.

Year	Operating days	Year	Operating days
1966	82	1974	320
1967	61	1975	420
1968	62	1976	540
1969	86	1977	700
1970	161	1978	880
1971	170	1979	1100
1972	200	1980	1380
1973	310		

These totals do not include fuel that will be discharged prior to 1966, which is estimated to represent 123 days of operating time.[2] It may be noted that the expected work load will exceed the NFS plant nominal capacity by about 1975.

An artist's concept and plan of the NFS plant[3] are shown in Fig. I-1. Design is complicated by the fact that the plant must be able to accept a wide variety of fuels of varying isotopic concentrations, including fully enriched uranium. The list includes uranium metal; uranium-molybdenum, uranium-Zircaloy, and uranium-aluminum alloys; ThO_2-UO_2; and slightly enriched UO_2 with a wide range of claddings and physical configurations. Sodium-bonded fuels and stainless-steel cermet fuels will not be accommodated, although provision for these may be added later.[2]

An unusual feature of the plant will be the use of mechanical head-end equipment for decladding and chopping the fuel into short lengths for leaching with nitric acid. Considerable development work has been done on this technique, but it has never been used in a chemical-processing operation. NFS has suggested a joint project with Oak Ridge National Laboratory (ORNL) to develop the head-end decladding equipment, but this matter has not yet been decided.[2]

In its application for AEC authorization to construct the plant, NFS presented the following information on the project's estimated capital requirements:[2]

Bechtel-constructed facilities		$23,085,000
NFS-provided related facilities		1,465,000
Plant site		500,000
NFS-construction related expenses		1,250,000
Working capital		1,100,000
Preoperational costs (startup, testing)		
Preoperational costs	$1,400,000	
Contingency for preoperational costs	2,000,000	3,400,000
Contingency fund (overall) available for construction		1,000,000
		$31,800,000

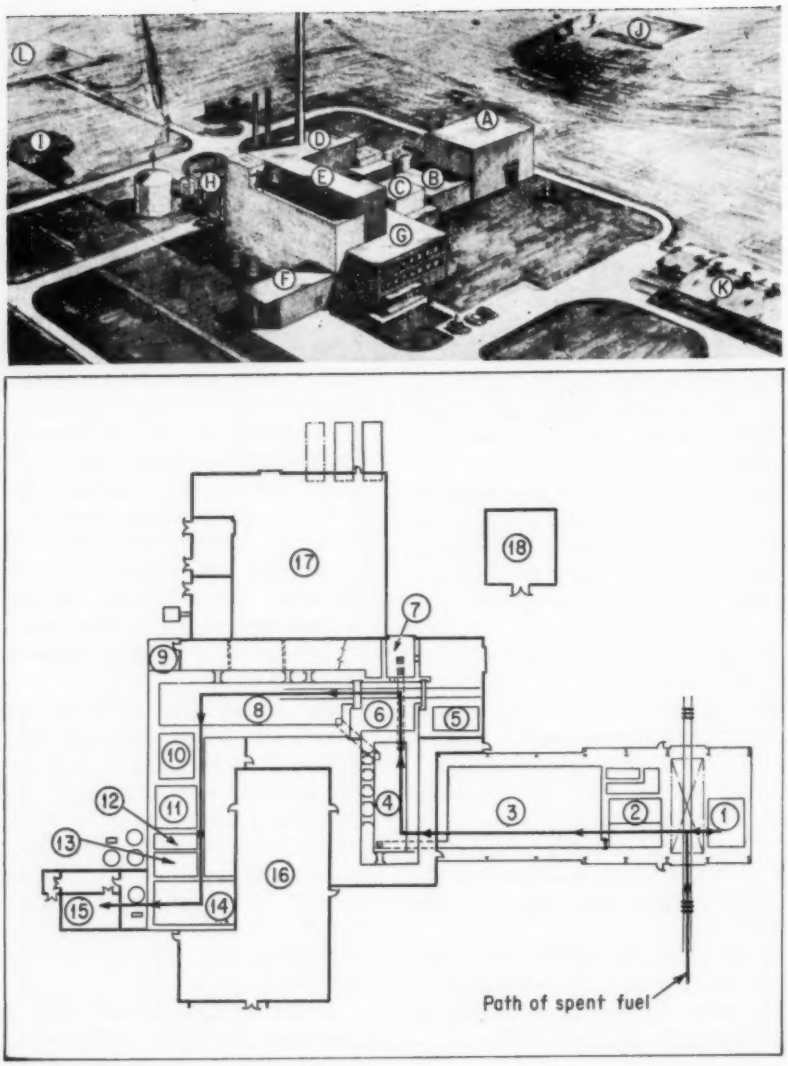

Fig. I-1 Fuel-processing facilities at planned NFS plant.[3] These facilities include: fuel-receiving building (A), containing cask decontamination area (1) and cask unloading pool (2); fuel-storage building (B), with water-filled pool for storage (3); process mechanical cell for decladding and chopping fuel (C, 4), surrounded by soaking pit (5), decontamination area (6), and scrap-removal facility (7); chemical-processing cell for fuel dissolution (D, 8), with associated acid recovery facility (9); process cells for solvent extraction and separation of plutonium and uranium (E, 10 to 14); product packaging and handling area (F, 15); control laboratories and office (G, 16) and utility building (H, 17). Also shown in latest artist's concept are: radioactive waste-storage tanks (I), storage lagoon for treated plant effluents (J), chemical and nonnuclear-materials warehouse (K) and low-level-waste burial area (L).

Foreign Plans

Three chemical-plant designers have been selected to prepare the preliminary design for Japan's first fuel-processing plant. Nuclear Chemical Plant, Ltd., of Britain will have primary responsibility but will be assisted by St. Gobain Nucléaire of France and Weinrich and Dart of the United States. The design will incorporate both French gaseous-waste-disposal technology and U. S. decladding techniques.

The plant is to have a capacity of 0.7 ton of natural-uranium fuel per day or its equivalent in enriched fuel. It is to be capable of handling the natural-uranium magnesium-alloy-clad fuel which will be used in Japan's first large nuclear plant, as well as Zircaloy-clad fuel enriched up to 3% and stainless-steel-clad fuel enriched up to 5%.

The present contract is for preliminary design only. New bids will be invited later for the detailed design.[4]

Economics of Fuel Processing

Commercial Prices for Refined UF_6

The production of refined uranium hexafluoride from uranium ore concentrates is currently being performed in the privately owned plant of Allied Chemical Corp. at Metropolis, Ill. The company has a contract with AEC for the processing of 5000 tons of U_3O_8 to UF_6 per year by a dry process based on chemical conversions in fluidized-bed reactors and purification of uranium hexafluoride by fractional distillation. This process differs substantially from that used in the AEC-owned plant at Weldon Spring, Mo., where purification of uranium is accomplished by aqueous solvent extraction prior to conversion to UF_6 at the AEC's Paducah, Ky., plant.

Reactor operators, anticipating private ownership of nuclear fuels in the future, are interested in knowing the prices that will be charged for production of pure UF_6 from ore concentrates. An AEC projection of costs is said to indicate a price of about $1 per pound of U_3O_8 processed, for government-owned plants in the future. The Allied Chemical price is now reported to be about $1.20 per pound; however, this price is expected to drop drastically after amortization of the plant is completed in March 1964. Allied's projection of future costs has not been released, but it is reported that the price is expected to be considerably below $1 per pound, but not below the AEC's incremental cost of processing additional material in the integrated Weldon Spring–Paducah plants.

Table I-1 EXPECTED NFS WORK LOAD AND COSTS OF PROCESSING

Type of material	Operating days	Basic and incremental processing	Escalation	Waste tanks	Total
Canadian reactor fuel	210.0	$ 4,948,388	$158,704	$ 306,810	$ 5,413,902
Low-enriched fuel	166.8	3,932,218	270,565	17,903	4,220,686
High-enriched fuel	248.2	5,842,752	298,783	2,565,333	8,706,868
Total	625.0	$14,723,358	$728,052	$2,890,046	$18,341,456

The AEC's five-year contract with Allied Chemical Corp. expires on Mar. 31, 1964. Renewal of the contract has not been decided, but consideration is being given to renewal on a one-year basis.[2,4]

The General Electric Company has been engaged in a study of some of the problems that may arise with the advent of private ownership of nuclear fuel materials and toll enrichment services in AEC gaseous diffusion plants. To carry out the study, General Electric Company has made several purchases of uranium ore concentrates in batches up to several hundred pounds from a variety of sources. The "yellow cake" was shipped to processing plants for conversion to UF_4 or UF_6. At least one batch was put through Allied Chemical's Metropolis plant. When the study is completed, it is intended to answer questions concerning procurement, licensing, accountability, analysis, and handling from the mill to the diffusion plant. The results show that natural uranium from a

choice of sources can be promptly available for toll enrichment in AEC diffusion plants.[2]

Financing Privately Owned Fuel Inventories

In 1962 the AEC submitted to the Joint Committee on Atomic Energy (JCAE) a proposal under which private ownership of nuclear fuels would be required after June 30, 1972. The increase in financing costs due to a change from government to private ownership and the nature of the conventional and alternative methods of financing which might be employed to finance privately owned fuel inventories have been examined by an *Ad Hoc* Committee of the Atomic Industrial Forum.[5] Since fuel-inventory charges are intimately associated with fuel-processing schemes—for example, their relation to the duration of the cooling time for spent fuel—the conclusions of the Committee may be of interest to the readers of *Reactor Fuel Processing.*

To illustrate the magnitude of the additional investment that would be required in nuclear plants in the event of a change to private ownership of nuclear fuel material, the following are the approximate costs of preparing a single loading of nuclear fuel material for a 450-Mw(e) water type reactor based on current prices:

1. To acquire 700 tons of U_3O_8, $11.2 million
2. To convert the 700 tons of U_3O_8 to pure UF_6, $1.5 million
3. To enrich UF_6 in the gaseous diffusion plant, $8 million
4. To convert enriched UF_6 to UO_2 and to fabricate and assemble fuel elements, $9 million

The cost of chemically processing one spent core would be about $3 million, and the cost of transportation to the processing plant would be about $0.5 million per core. The processing and transportation costs would be more than offset by the value of the spent fuel. On the basis of the July 1, 1962, AEC price schedule for enriched uranium, and $9.50 per gram for plutonium in the form of metal, the value of the unused uranium and plutonium recovered from the spent core would be about $11 million ($6.5 million of unused uranium and $4.5 million of plutonium).

The approximate time cycles required for the processing of nuclear fuel materials are also of interest in considering the magnitude of inventory charges. It would normally require:

1. About six to seven months' production of a large mill to produce the 700 tons of U_3O_8 required for the initial loading of a 450-Mw(e) water type reactor
2. Two weeks to a month to convert the ore concentrate to pure UF_6
3. Nine to twelve months to obtain the enriched UF_6 from the gaseous diffusion plant, to process it into UO_2, to fabricate the fuel elements, and to transport the fuel from the processing location to the reactor site

The storage of the fuel at the site and the initial loading of the fuel might consume two to three months. On the basis of a complex of technical and economic factors, the reactor operator might elect to run the reactor for two or three years and then replace the entire fuel load, or he might choose to replace a portion of the core at a time. The economics of the fuel cycle will probably encourage the replacement of a fraction of the core on an annual basis. The discharged, irradiated fuel would be allowed to cool for about four months before shipment to the processing plant. Transportation of the irradiated fuel could require several months, depending on the shipping distance and the number of available shipping casks. Four to eight months would be required to complete the processing of the entire core of a 450-Mw(e) water type reactor if it is assumed that the processing is to be done in a plant with a capacity of 1 ton/day.

The *Ad Hoc* Committee concerned itself only with financing the added cost attributable to a change from government to private ownership. The example used by the Committee shows that the value of such fuel material, exclusive of transportation and fabrication costs, could fluctuate from a low of $15 million to a high of $20 million after the nuclear fuel inventory reaches equilibrium. The Committee considered a number of familiar financing techniques and concluded that new, radical financing techniques need not be developed to accommodate a transfer of nuclear fuel material to private ownership. Some members of the Committee believe that, for an interim period in which only a small part of a utility company's facilities are nuclear, any increase in costs of financing nuclear inventories might be minimized by supplementary financing techniques. All members of the

Committee were of the view that the cost of financing such inventories will rise in the long run from the AEC use charge rate to the level of the composite cost of money to the reactor operator.

Fast-Reactor Processing Costs

General Electric Company's Vallecitos Atomic Laboratory has issued a report[6] that discusses the costs of processing irradiated fuel from a fast-neutron reactor fueled with uranium-plutonium oxides. The economic study is based on the use of a process involving an adaptation of the Hanford anion-exchange process[7] and the Idaho small-plant concept.[8]

Spent-fuel characteristics and processing-plant loads were based on a reactor concept for which the total electric power output is 517 Mw(e) and the burnup of the core amounts to 100,000 Mwd per ton of fuel. For each such reactor the spent-fuel load of both core and blanket amounts to 49 kg of uranium, plutonium, and fission products per day. The cooling time before processing was taken as 20 days.

The following reasons were given for not selecting a flow sheet based on solvent extraction: (1) the adverse effect of high radiation levels on solvent performance, (2) possible interface troubles from macro levels of fission products, and (3) the lack of incentive to recover uranium. If $5/kg is assumed as an approximate cost for depleted uranium dioxide, the price penalty for discarding the uranium was given as 0.003 mill/kw-hr.

It was assumed in the cost estimation that all costs except waste storage and product losses would be the same for an anion-exchange plant as those given for the Idaho small-plant concept based on solvent extraction. Scaling factors were assumed and used to estimate the variation in costs with plant throughput. The costs thus determined are given in Table I-2, and the unit processing costs are listed in Table I-3.

Table I-2 ESTIMATED COSTS FOR CONCEPTUAL PROCESSING PLANT[6]

No. of reactors Mw(e)	1 517	2 1035	3 1550
Annual costs, dollars			
Operating labor	238,000	425,000	511,100
Chemicals and expendable material	14,300	28,800	42,900
Utilities	5,100	7,300	11,000
Miscellaneous supplies	4,600	9,100	13,700
Fixed charges on investment	676,000	676,000	676,000
Total	938,000	1,146,200	1,254,700

Table I-3 ESTIMATED UNIT PROCESSING COSTS FOR CONCEPTUAL PLANT[6]

No. of reactors Mw(e)	1 517	2 1035	3 1550
Estimated costs			
Waste and product loss, mill/kw-hr	0.023	0.023	0.023
"Fixed" costs, mill/kw-hr	0.255	0.156	0.114
Total, mill/kw-hr	0.278	0.179	0.137
$/kg (U + Pu)	$80.50	$51.50	$39.40

Costs for Close-Coupled Type Processing

An initial study has been completed at Hanford comparing the conventional solvent-extraction processing costs with salt-cycle type, close-coupled fuel processing (see Sec. III) for a large [1000-Mw(e)] light-water-moderated reactor recycling self-generated plutonium.[9] Fuel exposure was taken as 14,500 Mwd/ton for each recycle step. Both stainless-steel and zirconium-clad oxide fuel elements and a broad range of cost inputs were considered. Plutonium was valued on the basis of average value of fissile plutonium as a replacement for U^{235} enrichment. Both lease and purchase of enriched uranium were considered. Input data included fuel-cycle losses, inventory periods, burnup, and unit costs.

For the reactor cases evaluated to date, a potential saving of 0.10 to 0.25 mill/kw-hr for salt-cycle type processing is indicated to be a reasonable possibility with plant capital cost limited to a range of $5 million to $10 million. This was considered to be a realistic capital cost target for the small (approximately 0.2 ton of uranium per day), simplified, single-purpose plant visualized. In other terms the savings ranged from $12 to $30 per kilogram of fuel processed or $700,000 to $1,750,000 per year for a 1000-Mw(e) reactor complex.

An economic study has also been made by Atomics International on the so-called AIROX process (reconstitution of fuel involving a pyro-oxidation-reduction step).[10] This approach to fuel processing is based on reenrichment and

remote refabrication of the spent fuel with very little removal of fission products. Cost penalties are imposed by reduced neutron economy; however, these added costs are said to be offset by the elimination of the shipping charges, the conversion charges, and the cost of expensive chemical processing.

Table I-4 COMPARATIVE FUEL-CYCLE COSTS[10]

[Uranium and Plutonium Recycle: (1) Reenrichment by Blending, (2) 15,000 Mwd/Metric Ton of Uranium Burnup per Cycle in a Boiling-Water Reactor, (3) Costs Calculated for Equilibrium Cycle (5th)]

Charges	AEC reference aqueous plant, mills/kw-hr	IDO on-site aqueous plant, mills/kw-hr	AIROX, mills/kw-hr
Burnup	0.716	0.716	0.867
Use	0.195	0.197	0.232
Loss	0.032	0.032	0.036
Conversion	0.137	0.137	0.022
Fabrication (Zircaloy-2 clad)	0.855	0.855	0.942
Shipping	0.202	0.073	0.015
Processing	0.213	0.491	Included in fabrication
Total	2.35	2.50	2.11

The studies were carried out for a 1000-Mw(e) complex, based on a boiling-water reference reactor. The results of the fuel-cycle cost comparison are presented in Table I-4. It was necessary to make the comparison over the whole fuel cycle in order to evaluate the effect of the processing method on all aspects of fuel-cycle costs. The results of the study indicate that a net saving of at least 0.2 mill/kw-hr is achieved with the AIROX process [about $1 million per year for 1000 Mw(e)].

Commercial Waste Disposal

Because of the establishment of land burial sites by private industry, the AEC withdrew from its "interim land burial" program for disposition of solid low-level radioactive wastes on Aug. 12, 1963. Land burial services for solid low-level wastes had been available to licensed users of nuclear materials at ORNL in Tennessee and at the National Reactor Testing Station (NRTS) in Idaho. Commercial burial sites have now been established by Nuclear Engineering Co., Inc., in Nevada and Kentucky on state-owned land. Burial charges established by Nuclear Engineering Co. are not to exceed 80 cents per cubic foot at the Kentucky site nor $1.50 per cubic foot at the Nevada site. Arrangements for additional privately operated disposal facilities are being completed in New York State at the site of the NFS plant. Principally because of economic considerations, more than 95% of the low-level wastes are now buried on land, the remainder being disposed of at sea.[11]

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Section

II

Reactor Fuel Processing

Research and Development on Aqueous Processing

Very little new information has been reported recently in the area of aqueous processing of reactor fuels. Mechanical preparation and electrolytic dissolution techniques continue to be of interest, and the understanding and applications of solvent extraction are broadening.

Preparation for Fuel Processing

Slansky[1] has recently presented a short survey of nuclear-fuel-processing methods and facilities, with particular emphasis on head-end techniques for the preparation of fuel for processing. The simplicity and multipurpose ability of chop-leach and electrolytic methods are noted, along with the limitations on the use of special dissolving reagents, such as sulfuric and hydrofluoric acids and aqua regia. A contribution from Dounreay[2] reviewed development work in England on the processing of irradiated fast-reactor fuels of various types which might possibly succeed the vanadium-bearing uranium-chromium alloy used in the first core for the Dounreay fast reactor. Uranium-plutonium fuel types considered included alloys (particularly those containing molybdenum), cermets, and ceramics (oxides and carbides). The process methods were largely similar to those developed in the United States. One somewhat novel technique was the volatilization of molybdenum as the oxide from uranium-molybdenum alloy in order to provide a separation of molybdenum from uranium and minimize the adverse effects of molybdenum in solvent-extraction steps. Good results were obtained in pure oxygen at a pressure of 0.2 to 0.5 mm and 1000°C, in that the molybdenum content of the U_3O_8 could be reduced to less than 0.1% by this means. A mechanical pretreatment method for use with stainless-steel-clad stainless-steel–uranium dioxide cermet fuels consisted of comminution of the fuel by milling with very light cuts to produce fine particles which could be leached with nitric acid. Leaching of 1-mil cuts of a cermet with 6*M* nitric acid dissolved more than 99.9% of the uranium.

Dissolution of Fuel Cores

In anticipation of processing of fuels containing both zirconium and zirconium dioxide, studies are being carried out at the Idaho Chemical Processing Plant (ICPP) on organic-based dissolution reagents that are capable of being used[3] in the range 100 to 170°C. This effort results from indications that zirconia is not attacked by aqueous fluoride solutions below 100°C, whereas it is attacked by molten ammonium bifluoride at about 200°C. Hot triethanolamine was found to dissolve ammonium bifluoride to a fluoride concentration of about 5*M* and to permit solution of at least 0.65*M* zirconium and 3 g of uranium per liter. Dissolution rates of zirconium and zirconia in this reagent will be investigated.

Total Dissolution of Metallic Materials

Studies at the ICPP on the effect of minor alloying elements on the rate of the mercury-catalyzed dissolution of aluminum in nitric acid have been noted previously.[4] Fletcher and others have recently summarized the work.[5] In tests with a dissolving reagent composed of 6*M* nitric acid containing 0.005*M* mercuric nitrate, copper and nickel ions in solution had a small inhibiting effect (less than a factor of 2) on the rate of dissolution of pure aluminum (99.999% aluminum), whereas the effects of dissolved uranium, or a simulated mixture of fission-product ions, was much less. In contrast, aluminum alloys containing a few percent copper, nickel, or

silicon dissolved in the mercury-bearing nitric acid at rates that were orders of magnitude lower than that of pure aluminum. Manganese, magnesium, and zinc in the alloys were found to have little effect on dissolution rate relative to that of pure aluminum; iron appeared to accelerate dissolution slightly. It was possible to overcome the inhibiting effect of silicon either by attaching a piece of pure aluminum to the alloy or by increasing the mercury-catalyst concentration from 0.005*M* to 0.05*M*, but results of this nature were not obtained with copper-bearing alloys.

Separations Processes

Solvent extraction and ion exchange continue to be the separation techniques of choice for the purification of solutions of irradiated fissionable materials. Currently, the bulk of the research work in solvent extraction involves determination of mechanisms, refinement of pertinent data, and development of applications to the recovery of fission products and transuranic elements. (Fission-product separations are discussed in Sec. IV of this issue.) Some studies are also being made of ion-exchange materials and applications.

A recent review article by Chernyaev and Ellert[6] discusses the significance of complexes of hexavalent uranium in all ways in which uranium is processed chemically in aqueous and organic liquid systems, including dissolution, precipitation, and extraction operations. Nitrate, sulfate, halide, and other systems are discussed. It is pointed out, for example, that generic relations among hydrated sulfate and nitrate salts can be indicated on the basis of the following series of formulas for the complexes:

Sulfates	Nitrates
$[UO_2(H_2O)_6]^{2+}SO_4$	$[UO_2(H_2O)_6]^{2+}(NO_3)_2$
$[UO_2(H_2O)_4(SO_4)]^{\circ}$	$[UO_2(H_2O)_5(NO_3)]^{+}(NO_3)$
$[UO_2(SO_4)(H_2O)_3]^{\circ}_n$	$[UO_2(H_2O)_4(NO_3)_2]^{\circ}$
$[UO_2(SO_4)(H_2O)_2]^{\circ}$	$[UO_2(H_2O)_2(NO_3)_2]^{\circ}_n$
$[UO_2(SO_4)(H_2O)]^{\circ}_n$	

The first three of these are cationic complexes, whereas the others are neutral. Those with a subscript "n" are presumably polymeric. It is proposed that the reactions leading to complex formation frequently involve a number of consecutive steps, the importance of which is not adequately indicated by the summary reactions as ordinarily written. In particular, the summary equation for peroxide dissolution is usually written as

$$UO_2 + 2H_2O_2 + 2NaOH = Na_2UO_5 + 3H_2O$$

whereas the formation of complexes with peroxo groups suggests that a series of reactions leading to formation of the $Na_4[UO_2(OO)_3]$ complex is more appropriate. Oxalate, carbonate, and fluoride complexes are similarly treated.

Solvent-Extraction Processes

Stewart[7] has recently summarized papers presented at the Transplutonium Element Symposium held at Argonne National Laboratory, May 15–17, 1963. The contributions included several papers on the separation of heavy elements by solvent extraction. Stewart notes that the trialkylamines are most useful in giving actinide-lanthanide separations, whereas the alkyl phosphoric acids give the best resolution of actinides from one another. Kosyakov of the USSR indicated that 0.5*M* diethylhexyl phosphoric acid in isooctane is adequately resistant to oxidizing agents such as permanganate, and to radiation, for high-alpha separations. The effects of alkyl group structure were also indicated. Branched chain substituents give much lower distribution ratios than those with straight chains. Increasing the length of the chain increases the distribution ratio slightly. Maly (of the USSR) described an interesting experiment using only 15 atoms of mendelevium to obtain the distribution coefficient of that element between tributyl phosphate (TBP) and 13.1*M* nitric acid by a reverse-phase chromatographic technique.

At the same meeting D. E. Ferguson and J. R. McWherter of Oak Ridge National Laboratory (ORNL) described the transuranium facility being constructed there and the processes to be used in it. Trilaurylamine (TLA) will be used for the transuranic extraction, and ethylhexyl phosphoric acid will be used for americium-curium isolation from lithium and aluminum chloride–salted chloride solutions. With the amine process, some difficulty has been encountered with separation from nickel, silver, and tellurium. Solvent exposure to 300 watts of alpha radiation per liter does not appear to be injurious to the processes, although its ef-

fect in producing destruction of hydrochloric acid at low acidity is a problem.

In other work on solvent extraction, Olander and Benedict[8] have continued work previously reported[9] on the mechanism of TBP extraction. In studying the process of nitric acid extraction by TBP, it was found that the kinetics were controlled by purely diffusional processes. The proposed mechanism involves transfer of nitric acid through the aqueous film, rapid complexing at the interface, and transfer of complex to the organic phase. Most of the resistance is encountered in the last step of the process.

Ion Exchange

Marchello and Davis[10] have reported studies of Dowex 1 exchange with a thorium anion complex in concentrated nitrate solution in an agitated bed. A method was developed for the prediction of the instantaneous loss curve in small pilot-plant beds from small-scale laboratory data. A time-dependent overall transfer coefficient was used.

A new type of ion-exchange material, potentially suitable for the extraction of uranium from ore leaches and other process solutions, was recently described by Degeiso and others.[11] This is a polymer prepared by heating resorcylic acid, formaldehyde, and resorcinol. A material having a working capacity of 3.4 millimoles per gram was obtained in preliminary studies.

Plant and Equipment Design and Operation

Processing Plant

Chemical Engineering reports on a nuclear-fuel-processing plant to be built and operated by private industry.[12] It is reported that Nuclear Fuel Services, Inc., a joint venture by Davison Chemical Company (78%) and American Machine & Foundry Atomics of Canada, Ltd., has obtained a construction permit.

The plant will be built near Buffalo, N. Y., at a cost of over $25 million. Nearly any type of spent nuclear reactor fuel can be handled in which the fuel proper can be reduced to a nitric acid solution. The initial overall capacity of the plant in terms of a single fuel having the indicated parameters will be as follows:

Quantity, tons/year	365
Burnup, Mwd/ton	20,000
Specific power, Mw/ton	27.5
Irradiation time, years	2
Load factor, %	85
Cooling time, days	150

The design of the plant itself is reported to make use of chiefly proven processes and procedures. The basic operation will utilize conventional Purex type solvent-extraction processes, which have been used full scale for more than a decade.

The proposed plant is described in some detail. Fuel will be stored under water and conveyed to a mechanical cell, where hardware can be removed through the use of manipulators, saws, etc. A hydraulic-ram-driven shear cuts the fuel into short lengths (similar to the shear developed at ORNL and described in several past Reviews). The dissolvers are heated stainless-steel tanks contained in a remotely maintained cell. Off-gas scrubbers and iodine-removal equipment are included. Solvent extraction is accomplished by seven 10-in.-diameter pulse columns and three smaller ones for plutonium. The TBP-kerosene solvent is recovered and purified for reuse. Nitric acid can be recovered from waste evaporators. Waste will be stored in large tanks that are carefully monitored for leaks. Rodger reports[13] that, even if the double tanks should leak, the waste will not reach the water table because of a nearly impervious layer of soil in the area. (For additional information on the NFS plant, see Sec. I of this issue.)

Slurry Pumps

Bailey presents a comprehensive report[14] on tests of pumps to be used for pumping corrosive, erosive liquids. The tests were made to

Table II-1 EROSION RATE OF SUSPENDED-SHAFT PUMP IN ACID-ALUMINA SERVICE[14]

Location	Erosion rate, mils/year	
	Initial 900-hr test with 100 g of undissolved solids per liter	Subsequent 1900-hr test with average of 300 g of undissolved solids per liter
Impeller suction	357	36
Impeller	Not measurable	9
Suction intake	570	104
Casing at top of volute	179	76

Table II-2 BEARING EROSION RATES[14]

Item	Erosion rate, mils/year: Alumina bearings A*	Alumina bearings B†	Boron carbide and stainless-steel‡ bearings
Suction bowl bearing	3.05	1120	2.08
First-stage bowl bearing		654	1.69
Second-stage bowl bearing		626	2.47
Shaft sleeve in suction bowl	3.05	500	2.21
Shaft sleeve in first-stage bowl			1.56
Shaft sleeve in second-stage bowl			1.56
Shaft sleeves is discharge head	0.76	523	1.82

*A, pump operated 1152 hr recirculating an alumina-water slurry containing 62 g of undissolved alumina per liter.

†B, pump operated for two weeks recirculating a 1.0*M* HNO_3 slurry containing 2 to 20 g of undissolved alumina per liter.

‡Pump operated 3400 hr recirculating a 1.0*M* HNO_3 slurry containing 90 g of undissolved alumina per liter. The bowl bearings were made of boron carbide, and the shaft sleeve bearings were made of hardened stainless steel type 440-C.

determine the feasibility of using the pumps for pumping nitric acid–alumina slurry in the waste calciner facility at the ICPP; however, some of the test procedures and conclusions could be applied to other systems. He reports that both a suspended-shaft pump with non-wetted bearing and a turbine type pump with hardened bearing could be used for this particular service. For the turbine pump, boron carbide was recommended for the bowl bearings, and, for the shaft journals, type 440-C hardened stainless steel was recommended.

Table II-1 shows the results of tests for the suspended-shaft centrifugal pump. Table II-2 shows the tests on the turbine pump with two bearing arrangements. Detailed information on test procedures is given in the report.

Solvent-Extraction Contactors

Woods et al.[15] present an extension of the work on hydroclones previously reviewed.[16] This program is aimed at the development of a solvent-extraction contactor having a high ratio of throughput to holdup in order to minimize solvent damage from radiation. In the current work a variety of configurations were tried, and the data were presented in detail. The runs were made using aqueous uranyl nitrate salted with 1*M* sodium nitrate and 18% TBP in Amsco. Four hydroclones (Mark I design) were used with the organic solvent entering into the feed to the fourth clone. Below these four clones, there were three clones of the Mark IV design which served as polishers to return entrained organic.

Figures II-1 to II-3 show plots of the results (inset shows the variable studied). Figure II-1 shows the effect of increasing the clearance at the outer ring of the skirt. As this restriction was opened, the capacity dropped slightly at first, then increased to a maximum at a clearance which should offer negligible pressure drop, and then decreased slightly for further enlargement. The efficiency of the clones is seen to rise from 40 to 78% where it appears to hold constant. Figure II-2 shows the effect of increasing the volume in the chamber defined by the underflow skirt without increasing the

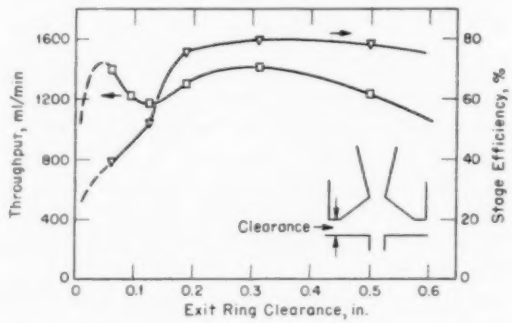

Fig. II-1 Effect of clearance in flow channel out of the underflow skirt chamber: Mark I stacked clone contactor.[15]

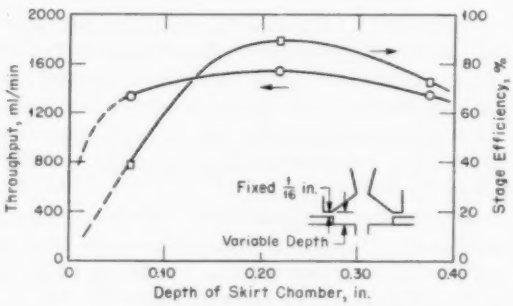

Fig. II-2 Effect of underflow skirt chamber volume: Mark I stacked clone contactor.[15]

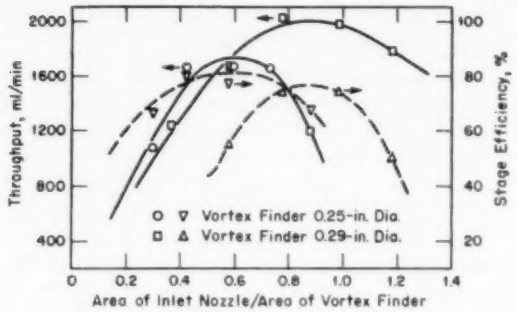
Throughput, ml/min
Stage Efficiency, %
Area of Inlet Nozzle/Area of Vortex Finder
O ▽ Vortex Finder 0.25-in. Dia.
□ △ Vortex Finder 0.29-in. Dia.

Fig. II-3 Effect of feed nozzle size: Mark I stacked clone contactor.[15]

dimension of the flow restriction at the outer rings. Again it is noticed that the capacity increases slightly and that the stage efficiency increases markedly from about 40 to about 80%. From these studies it must be concluded that adequate time of contact either in the vortex finder or in the underflow skirt chamber is important, as also is the provision of flow restriction to assure symmetrical radial flow.

The results of a study to explore the effect of the size of the inlet nozzle to the clones are shown in Fig. II-3. As the size of the channel is increased by a factor of 2 over the initial design, both the capacity and the efficiency increase to a maximum beyond which velocities are inadequate to properly drive the clone.

Culler[17] further reports that a new design series (Mark X) of contactor has been run on the same system giving a "performance factor" (throughput times efficiency) of 2.7 to 2.9 vs. 2.3 for the arrangements previously used. He reports that the new design has features that were suggested by previous tests: an underflow skirt, a mixing chamber between the underflow part of one stage and the vortex finder of the stage below, and a baffled passage between the mixing chamber and the pump suction plenum designed to promote radially symmetrical flow.

Electrolytic Dissolvers

Work on electrolytic dissolution systems has been reported[18–20] several times in this Review. However, the following two reports are worthy of note. A pilot model dissolver that is to be

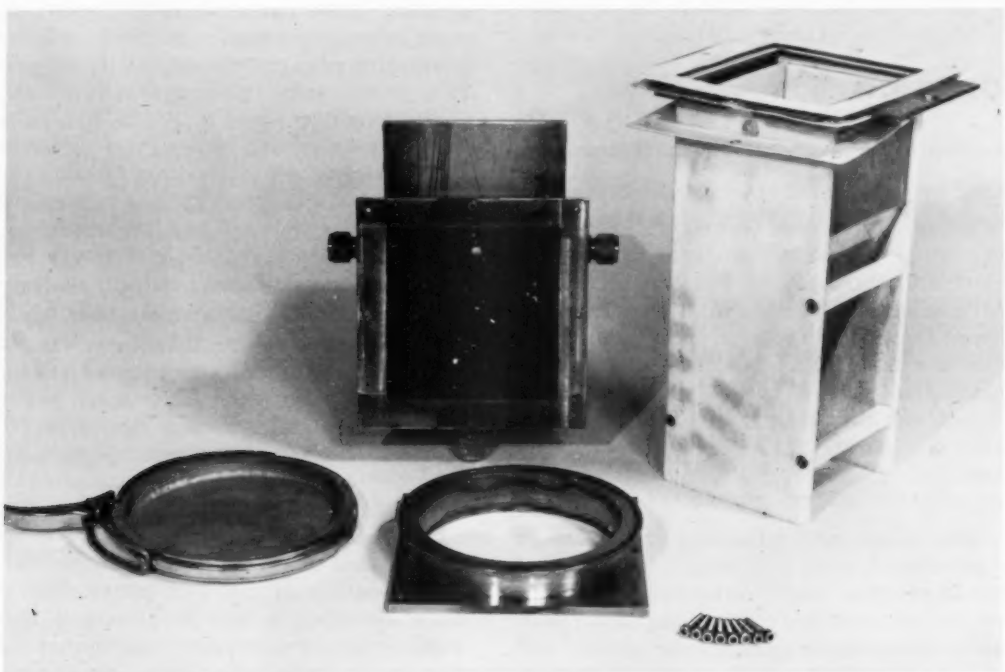

Fig. II-4 Experimental solution-contact electrolytic dissolver components.[3]

used at the ICPP is described by Weech.[3] This dissolver is a solution contact type, in which the fuel pieces are separated from the electrodes with the current entering indirectly through the electrolyte. (The model, however, is convertible to the metal-to-metal contact type, in which the fuel is in direct contact with the anode.) The dissolver, which was designed as a short section of an otherwise full-scale horizontal dissolver, can accommodate 4-in. lengths of full-size fuel elements of the Stationary Medium Power Plant (SM-1), Organic-Moderated Reactor Experiment (OMRE), or Heat-Transfer Reactor Experiment (HTRE) type. The material is 304L stainless steel with Teflon-insulated electrodes. The anode is $\frac{1}{8}$-in.-thick platinum-plated titanium, and the cathode is 0.010-in.-thick platinum. Figure II-4 shows a photograph of the parts. Under special conditions, current densities as high as 3100 amp/sq ft (anode facing) and 6000 amp/sq ft (cathode facing) were observed.[21] However, some improvements are being made to the system.

The second report is a final report[22] on bench-scale work on metal-to-metal electrolytic dissolution covered in part by a previous Review.[20]

Corrosion

Corrosion in Sulfex- and Zirflex-Process Media

Most of the power-producing nuclear reactors, either in use or proposed, utilize fuel elements that are clad in stainless steel or in a zirconium alloy. Several AEC sites have developed a variety of chemical procedures for the removal of these cladding materials. Two of the most promising procedures are the Sulfex process, for the decladding of stainless-steel fuels, and the Zirflex process, for the decladding of zirconium-clad or zirconium-alloy-clad fuels.

ORNL has recently demonstrated both of these procedures in a hot cell using fuels irradiated up to 28,200 Mwd/ton.[23] Decladding for the Zirflex process consisted of dissolving the Zircaloy jackets in boiling $6M$ ammonium fluoride–$1M$ ammonium nitrate, and the Sulfex decladding step consisted of dissolving the stainless-steel jackets in refluxing $4M$ sulfuric acid. Some corrosion information that was accumulated during these tests is reported below.

During the Zirflex test, Nionel corrosion specimens accumulated about 47 hr of exposure in the totally immersed condition while held in contact with the dissolving Zircaloy-clad specimens, plus an additional 14.5 hr in the boiling core dissolvent ($4M$ nitric acid). On the basis of weight loss of the Nionel coupons, the calculated corrosion rate was 2.8 mils/month; this is considered to be a satisfactory rate.

Weight gains during the Sulfex portion of the program prevented the collection of accurate data on the behavior of the specimens. No severe localized corrosion was noted, however.

Construction Materials for ORNL Transuranium-Element Processing Plant

Plans are progressing for the isolation and recovery of transuranium elements at ORNL. It is planned to utilize a process which makes use of concentrated chloride solutions covering a wide range of acidities, some of which will contain nitrate. In solutions containing appreciable concentrations of the transuranium elements, peroxide is expected to be present as a product of the radiolysis of the water. In the most recent corrosion tests conducted on this processing system,[24] the sensitivity of tantalum to hydrogen pickup in a number of Transuranium Processing Facility (TRU)-related environments (solution and vapor) was investigated. Included among the tests were tantalum-plated specimens and tantalum specimens coupled with aluminum specimens. The latter simulate a condition in which aluminum-clad target rods in contact with a tantalum vessel will undergo dissolution in hot hydrochloric acid solution.

Some of the results obtained are shown in Table II-3. With the exception of the plated specimens, corrosion rates were negligible (hydrogen pickup was less than 3 ppm). The plated specimens picked up large quantities of hydrogen owing to complete dissolution of the stainless-steel base metal. The dissolution resulted from contact with the hot $6M$ hydrochloric acid solution through defects in the 3- and 10-mil-thick tantalum plates. The latter were extremely brittle after completion of the test. Some hydrogen pickup also occurred in the tantalum specimens that were coupled with type 1100 aluminum; after they were corrosion tested, the specimens were bent 180°, but no

Table II-3 HYDROGEN-PICKUP TESTS WITH TANTALUM* IN TRU-RELATED ENVIRONMENTS[24]

Environment		Time, hr	Hydrogen content, ppm	
			Solution	Vapor
Oxygenated 3*M* HCl at 105°C		1000	<1	<1
Oxygenated 6*M* HCl at 43°C		1000	3	
Oxygenated 6*M* HCl at 105°C		1000	<1	<1
Oxygenated 10*M* LiCl–0.1*M* HCl at 130°C		1000	2	<1
Oxygenated 10*M* LiCl–0.1*M* HCl–0.0023*M* $LaCl_3$ at 130°C		1000	<1	<1
H_2O_2-containing 10*M* HCl–0.0023*M* $LaCl_3$ at 35°C		1000	<1	<1
Coupled with type 1100 aluminum in 6*M* HCl at 105°C	(1)	7	64	
	(2)	6.5	54	
Purposely damaged tantalum plate in oxygenated 6*M* HCl at 105°C	(1)	100†	3100	
	(2)	350‡	2000	

*Unexposed control material was found to contain between 1 and 2 ppm of hydrogen.

†Three-mil-thick defected plate; stainless-steel base metal dissolved completely during test.

‡Ten-mil-thick defected plate; stainless-steel base metal dissolved completely during test.

signs of cracking which would indicate embrittlement were observed.

In a second investigation directed toward TRU-related environments, welded and unwelded Hastelloy C was exposed to 6*M* hydrochloric acid solution at 35°C. Three variations in exposure conditions were utilized: (1) no added oxidant, (2) oxygen aeration of the test solution, and (3) continuous addition of hydrogen peroxide to the test solution. Total exposure time[24] was 1000 hr, with solutions being replaced each 100 hr. In the absence of an added oxidant, the corrosion rate in solution was appreciably less than the rates obtained in tests with either oxygen aeration or peroxide addition. The rates observed were 0.7, 5.2, and 4.0 mils/month, respectively, for these environments. Weight loss vs. time of exposure curves indicate reasonably uniform corrosion for the solution containing no oxidant. In the case of the oxygen-aerated and hydrogen peroxide-containing environments, a 50% increase in the slopes occurred between the beginning and end of the tests.

All specimens underwent preferential attack in heat-affected zones adjacent to welds, with the most severe attack taking place in the oxygen-aeration test. The measured depth of penetration of the preferential attack varied between 28 and 32 mils. Careful examination of the data presented in the report confirms the presence of the preferential attack.

The problem of maintenance in the transuranium processing plant is a serious one, and it is proposed to provide the capability of removing entire equipment pieces or particular defective components from the plant. Quick-acting fluid disconnects will be placed in strategic locations to permit the remote disassembly of the various process lines necessary for the substitution of a new equipment component. The disconnects will utilize metal-to-metal seats for sealing action and will probably be made from either type 304 stainless steel or Hastelloy C. In order to ensure long-term integrity of these disconnects, it is necessary to demonstrate that their corrosion rates in process environments were acceptably low. Toward this end, accelerated tests are being conducted in aqua regia.[15] Results are given for Hastelloy C corrosion, which was found to be serious at welds. When Hastelloy C was given an unspecified heat-treatment for the purpose of eliminating the high rate of corrosion at weld areas, the sealing qualities of the disconnect were destroyed. Alternative methods of solving this corrosion-sealability problem are being investigated.

Corrosion of Stainless Steel in HNO_3 Containing Dissolved Chromium

Several sites, notably Savannah River and the ICPP, have been concerned over the accelerated corrosion that occurs when austenitic stainless steels are exposed to refluxing nitric acid which has been contaminated with accumulated stainless-steel corrosion products, particularly chromium. Additional significant observations on this subject have been made by several French experimenters.[25,26]

Construction Materials for the Electrolytic-Dissolution Process

Decker of the ICPP completed a study[27] in which he exposed several plastic materials and one alumina ceramic (AP-35) to solutions characteristic of the electrolytic dissolution process[28] while they were simultaneously exposed to high gamma-radiation fields. The solution used for the initial screening tests was 1*M* nitric acid containing 75 g of dissolved stain-

less steel per liter. New samples of those materials which were resistant to the solution were then exposed simultaneously to the boiling dissolver-product solution and a gamma-radiation field. Criteria used for the evaluation of these materials after exposure to the test solution and gamma radiation included the impact strength, compressive strength, and electrical resistivity. According to the author, the most favorable performances were with Kynar (polyvinylidene fluoride resin), up to 1×10^9 r total dose; Marlex (high-density polyethylene), up to 2×10^9 r total dose; G-7 glass fabric silicone resin laminate, up to 1×10^{10} r total dose; and ceramic alumina (AP-35), greater than 1×10^{10} r total dose. The report includes detailed data on the mechanical and electrical properties measured and photographs which show the visible effects.

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Section

III

Reactor Fuel Processing

Research and Development on Nonaqueous Processing

Volatility Processes

Liquid-Phase Processes

Nitrofluor Process. The initial step in the Nitrofluor process, which is being developed by Brookhaven National Laboratory (BNL), is the dissolution of reactor fuel assemblies in an anhydrous NO_2-HF solution. A solution that contains 20 mole % NO_2 in HF is capable of dissolving a wide variety of fuel-element materials at satisfactory rates over a temperature range of 100 to 200°C. After dissolution of the fuel elements, uranium and plutonium are recovered as volatile hexafluorides. An alternative solvent, NOF·3HF, is being evaluated since it offers the advantage of being less corrosive and not as highly oxidizing as the NO_2-HF solvent. Another advantage results from the fact that the boiling point (95°C) of NOF·3HF is 40°C higher than that of 20 mole % NO_2–80 mole % HF solution, thereby permitting fuel dissolution at considerably lower operating pressures.

Uranium dioxide dissolves in NOF·3HF to produce nitrosylium hexafluorouranate ($NOUF_6$) which is soluble in the solvent. The rate of dissolution of sintered uranium dioxide fuel pellets in NOF·3HF has been measured at BNL.[1] It was observed that high dissolution rates for uranium dioxide [51 mg/(cm^2)(min)] are achieved in the presence of a large excess of the solvent in which the $NOUF_6$ reaction product is completely soluble. Analyses of the gases produced in the dissolution of uranium dioxide in NOF·3HF have indicated that the gaseous products are NO and NO_2.

Uranium is recovered from solid $NOUF_6$ by converting $NOUF_6$ to a volatile uranium compound.[1] The method involves contacting the solid with gaseous NOF·3HF or NOF at 400 to 500°C to form a volatile uranium compound. Complete uranium volatilization from $NOUF_6$ has been demonstrated in laboratory tests. Uranium tetrafluoride has been identified as one of the solids that results from the decomposition of the volatile compound in a downstream zone maintained at 350°C. This method has been extended to include volatilization of uranium from several other source materials. It was demonstrated that UF_4, UO_2F_2, and UO_2 are completely converted to a volatile compound at 400°C in the presence of gaseous NOF. Although the reactions are not yet understood, this method appears to have direct application to the Nitrofluor process. Thus NOF·3HF could serve initially as the fuel solvent and subsequently as the volatilizing agent. This use of NOF·3HF has been tested for the processing of high-zirconium alloy fuels containing enriched uranium. The alloy was first dissolved in NOF·3HF at 80°C. The bulk of the zirconium precipitated as an insoluble complex salt. Since the concentration of uranium does not exceed the solubility of $NOUF_6$, the uranium remains in solution. After dissolution, the excess solvent was evaporated at 66°C and condensed in a cold trap. The zirconium-uranium salt residue was heated to 460°C for 2.5 hr in the presence of flowing NOF·3HF vapor (90 mm Hg abs. pressure) from the condensed dissolver solution. Approximately 80% of the uranium volatilized from the residue and was collected in a downstream condenser maintained at a temperature of 250°C. Less than 10 ppm of zirconium accompanied the volatile uranium. Further work on this volatility method is planned to demonstrate the complete removal of uranium from systems containing large quantities of zirconium.

Dry Halogenation Pocesses

Several processes that involve separation methods based on the relative volatility of chlo-

rides are being developed for the processing of reactor fuel elements. Studies are currently being made on zirconium and molybdenum alloy fuels containing enriched uranium. Initial separation of uranium from the alloy is achieved by converting the alloying metal to a volatile chloride ($ZrCl_4$) or oxychloride ($MoO_2Cl_2 \cdot H_2O$). Final recovery of uranium is achieved by fluorination to produce volatile uranium hexafluoride. Processes based on the fractional sublimation of chlorides are also being developed for ceramic fuels of low uranium enrichment.

Zirconium-Uranium Alloy Fuels. Engineering-scale studies are continuing at Argonne National Laboratory (ANL) on a process for the recovery of highly enriched uranium from zirconium alloy fuels.[2] The process is based on the use of hydrogen chloride, at a temperature of 400°C, to convert zirconium-uranium alloy to volatile zirconium tetrachloride and nonvolatile uranium trichloride. Recovery of uranium is achieved by the reaction between uranium trichloride and fluorine at 360 to 500°C to produce volatile uranium hexafluoride. Both reactions are conducted in a fluidized bed of inert Alundum (aluminum oxide) particles.

Process studies are being conducted in a 1½-in.-diameter fluid-bed reactor. Since knowledge of the hydrochlorination rate and gas composition is essential to the design and successful operation of the process equipment, the amount of hydrogen produced during the reaction of HCl with the alloy is measured continuously by means of a thermal conductivity cell.[2] From analyses of the data, it appears that the hydrochlorination step can be divided into two parts—a constant rate period and a falling rate period. During the constant rate period, the reaction in the fluidized bed is self-sustaining, and steady state may be attained for a given flow of inlet gas. Under these circumstances the efficiency of hydrogen chloride utilization is constant. During the falling-rate period, the reaction in the fluidized bed is not self-sustaining, and the temperature of the bed has to be maintained either by increasing the concentration of hydrogen chloride in the inlet gas stream or by supplying heat to the reactor. The reaction rate and efficiency of hydrogen chloride utilization both decrease rapidly during the falling-rate period.

Recent studies[2] have involved the successive hydrochlorination of several batches of alloy, followed by a single fluorination. A fresh charge of alloy was added to the reactor soon after the constant-rate period for the preceding batch had ended. Performing most of the hydrochlorination during the constant-rate period greatly improved the overall efficiency of hydrogen chloride utilization as compared to tests employing single charges. Results from the multiple hydrochlorination tests showed that a significant saving in processing time and reagents can be achieved by the use of this process cycle instead of performing both hydrochlorination and fluorination steps for individual batches.

The volatile zirconium tetrachloride produced during hydrochlorination of uranium-zirconium alloy fuel represents a waste stream that requires processing for convenient storage. Zirconium tetrachloride is rapidly converted to solid zirconium dioxide by a gas-phase reaction with steam. Process studies are being made at ANL[2] to demonstrate this reaction in a 6-in.-diameter fluid-bed system. Satisfactory operation has been demonstrated over the following ranges of conditions:

1. Bed temperatures: 250 to 500°C
2. Average particle sizes of starting beds: 60, 90, and 120 mesh
3. Inert-bed materials: zirconium dioxide, sand, and Alundum
4. Zirconium tetrachloride feed rates: up to 3.25 kg/hr
5. Run durations: up to 11.5 hr

Tsirel'nikov and associates[3] have investigated the corrosion of zirconium tetrachloride vapors on 1Kh18N9T steel* and nickel at high temperatures. It was observed that the rate of corrosion of 1Kh18N9T steel increased with an increase in temperature and zirconium tetrachloride pressure. The rate of corrosion of steel in zirconium tetrachloride vapor was observed to be a linear function of time with representative rates of corrosion being 0.36 g/(m²)(hr) at 350°C and 0.42 g/(m²)(hr) at 450°C. The corrosion of nickel obeys a logarithmic law which suggests the formation of a protective film of nickel chloride on the surface of the metal.

Molybdenum-Uranium Alloy Fuels. Workers at the Department of Plutonium, C.E.A. Fontenay-aux-Roses (France), have developed

* 1Kh18N9T steel has a composition of 18% chromium, 9% nickel and is tantalum or titanium stabilized; this is equivalent to type 321 or 347 stainless steel.

a method for processing molybdenum-uranium alloy fuels based on the volatility of molybdenum as an oxychloride or oxyfluoride.[4,5] Laboratory tests have shown that uranium-molybdenum alloys are attacked by gaseous mixtures of hydrogen chloride and oxygen or hydrogen fluoride and oxygen at temperatures of about 300°C.

The reaction of the alloy with mixtures of hydrogen chloride and oxygen leaves the uranium in the form of a nonvolatile residue, mainly U_3O_8, and a large part of the molybdenum is volatilized as the molybdenum oxychloride ($MoO_2Cl_2 \cdot H_2O$). Alloy penetration rates of 1.5 mm/hr at 450°C and 2.7 mm/hr at 575°C were obtained with an equimolar mixture of oxygen and hydrogen chloride as the reacting gas. The reaction rate was observed to increase slightly with increasing oxygen concentration in the reacting gas mixture. Approximately 90% of the molybdenum was removed from the alloy as volatile $MoO_2Cl_2 \cdot H_2O$ at a reaction temperature of 485°C. The remainder of the molybdenum appeared to be unrecoverable by this method. Less than 0.6% of the uranium was carried over with the volatile molybdenum oxychloride.

Regardless of the molybdenum content of the alloy, considerable attack by mixtures of hydrogen fluoride and oxygen was observed at temperatures as low as 250°C. The reaction with $HF-O_2$ mixtures leaves uranium in a form of a nonvolatile residue, the composition of which varies with the concentration of hydrogen fluoride in the reacting gas mixture. As the hydrogen fluoride concentration increases, the amount of uranium tetrafluoride in the residue increases, the fraction of U_3O_8 decreases, and the concentration of UO_2F_2 passes through a maximum of 50 wt.% at a hydrogen fluoride concentration of approximately 50 vol.%. The ratio of UO_2F_2/UF_4 in the residue varies, depending on the concentration of hydrogen fluoride in the reacting gas, and is less than the ratio of 2 obtained by the reaction between U_3O_8 and hydrogen fluoride. Therefore it may be assumed that the oxidation product of uranium is not entirely U_3O_8 but may contain UO_2. At a hydrogen fluoride concentration of 67 vol.%, the uranium residue contains approximately 50 wt.% UF_4 and 50 wt.% UO_2F_2.

A molybdenum-uranium alloy penetration rate of 1.1 mm/hr was obtained regardless of the initial reaction temperature and gas composition. This alloy penetration rate corresponds to a reaction rate of 1.75 g/(cm²)(hr). These data may reflect the fact that the alloy sample temperature in most tests reached 600 to 680°C during the reaction period even though the operations were conducted at various initial temperatures. The rate of reaction increased slightly with an increase in molybdenum concentration in the alloy. The molybdenum in the reaction product appears as a volatile oxyfluoride, the composition of which has been estimated to be $Mo_2O_3F_4$. The percentage of the molybdenum converted to the volatile oxyfluoride increases with reaction time and attains a value of 80% after 6 hr. Less than 0.1% of the uranium is lost in the exit gas stream.

The process utilizing $HF-O_2$ mixtures offers the advantage over the process in which $HCl-O_2$ mixtures are used since the partially fluorinated uranium product which is obtained can lead to significant savings in reduced fluorine requirements for the subsequent recovery of uranium as volatile uranium hexafluoride.

Corrosion tests have also been made[4] to evaluate possible construction materials for use in the processing of molybdenum-uranium alloy fuels. Two series of tests were made: (1) alternating exposure periods of 24 hr each with fluorine followed by a mixture of 80 vol.% hydrogen chloride and 20 vol.% oxygen were used for 5 cycles, and (2) alternating exposure periods of 24 hr each with fluorine followed by an equimolar mixture of hydrogen fluoride and oxygen were used for 10 cycles. The metal specimens were exposed at 400°C in both series. In the first series of tests, Hastelloy F, QA-18, QH-76, Inconel, Ancorros cupronickel, and NP cupronickel showed excellent resistance to corrosion. However, nickel did not exhibit good corrosion resistance. In the second series of tests, Monel, Inconel, and the cupronickel alloys showed excellent resistance to corrosion. Hastelloy B and the alloys QA-18 and QH-77 were badly corroded, whereas Hastelloy F and Hastelloy 20-25 were completely fluorinated. The results of the two series of tests are presented in Table III-1.

Processes Based on Uranium Chlorides. J. Schmets and associates at Centre d'Étude de l'Énergie Nucléaire (CEN) at Mol, Belgium, are developing a method for processing irradiated uranium and uranium compounds based on the fractional sublimation of chlorides. Recent work[6,7] has been concerned with the processing of uranium dioxide fuel. The proposed method for processing UO_2 fuel involves two major

Table III-1 CORROSION TESTS OF ALLOY SPECIMENS AT 400°C WITH ALTERNATING CYCLES OF FLUORINE AND HCl-O_2 MIXTURE OR FLUORINE AND HF-O_2 MIXTURE[4]

(Test Conditions: Series 1, Alternating Exposure Periods of 24 Hr Each with Fluorine Followed by a Mixture of 80 Vol.% HCl–20 Vol.% O_2 for 5 Cycles; Series 2, Alternating Exposure Periods of 24 Hr Each with Fluorine Followed by a Mixture of 80 Vol.% HF–20 Vol.% O_2 for 10 Cycles)

Alloy	Composition of alloy, wt.%						Penetration, mm/year	
	Fe	Cr	Ni	Cu	Mo	Other	Series 1	Series 2
QA-18		20	80				0.025	1.4
QH-76	5	16	63		16		0.08	
QH-87	5		67		28			0.3
Hastelloy F	23	22	45		6		0.01	7
Hastelloy 20-25	3	20	10			Co(67)		7
Hastelloy B	5	1	66		28			1.5
Nickel			100				0.7	
Monel			60–70	25–35			0.01	0.00
Inconel	6	14	80				0.00	0.00
Cupronickel Ancorros			69	29		Mn(2)	0.06	
Cupronickel NP			69	29		Mn(2)	0.09	0.01
Cupronickel UN26Z17			69	29		Mn(2)		0.04

steps: (1) oxidation of uranium dioxide to U_3O_8 and (2) chlorination of U_3O_8 to UCl_6 by a gas-phase reaction with a mixture of carbon tetrachloride and chlorine. Preliminary tests[6] have shown that the rate of reaction between UO_2 or U_3O_8 and mixtures of carbon tetrachloride and chlorine are nearly identical at 350°C. However, the chlorination reaction with U_3O_8 appears preferable for use in the process scheme since UCl_5 and UCl_6 are produced directly in the reaction. A relatively large fraction of the product resulting from the chlorination of UO_2 appears as UCl_4 and UO_2Cl_2, which must be further chlorinated to UCl_6 in a subsequent step.

Laboratory tests[6] have shown that the rate of chlorination between U_3O_8 and a gaseous mixture of carbon tetrachloride and chlorine can be increased significantly by the presence of certain inorganic chloride salts. The reaction is accelerated by $MgCl_2$, $CdCl_2$, $NiCl_2$, $BaCl_2$, and $CaCl_2$, whereas $SrCl_2$, $ZnCl_2$, LiCl, and CsCl appear to inhibit the chlorination reaction.

Studies are also being made at CEN[6,7] to determine if various fission-product materials can be separated from uranium during the chlorination step. Two methods are under investigation and involve the formation of fission-product chloride double salts. Certain fission products, such as zirconium and niobium, form volatile chlorides ($ZrCl_4$, $NbCl_5$) which are not easily separated from the volatile uranium chlorides formed in the process. These fission-product chlorides, however, do form double salts with certain inorganic chlorides which exhibit negligible vapor pressures even at high temperatures. This property of the double chloride salts offers a means for decontaminating uranium chlorides. Two schemes for decontaminating uranium are being investigated. In one scheme the irradiated U_3O_8 is mixed with a suitable inorganic chloride salt, and the mixture is chlorinated in a gas-phase reaction with a mixture of carbon tetrachloride and chlorine so as to produce volatile UCl_6 and concurrently fix the fission products in double chloride salts. In the other scheme the contaminated uranium hexachloride, which is produced in the chlorination of U_3O_8, and the volatile chlorides of the fission products are sublimed through a layer of inorganic chloride salts in such a manner as to fix the fission products as double salts but without fixing the uranium.

Direct Fluorination Process

A direct fluorination volatility process is being developed at ANL[2] to recover uranium and plutonium from spent uranium oxide fuel elements that are clad with Zircaloy or stainless steel. The proposed process involves decladding by an appropriate reaction in a fluidized bed. Plutonium hexafluoride and uranium hexafluoride, which are formed upon reaction of the declad oxide fuel with fluorine, may be separated

from volatile fission products by distillation and then may be separated from one another by thermal decomposition of plutonium hexafluoride.

Direct Fluorination of Uranium Dioxide Pellets. The direct fluorination of uranium dioxide pellet fuel is being studied at ANL[2] on an engineering scale in a 3-in.-diameter air-cooled reactor with the aim of optimizing reaction rate, fluorine efficiency, and fluidization quality. The system used in the studies consists of pellets in the form of a packed bed, with inert solid (Alundum) fluidized in the space above the pellets and in the voids of the pellet bed. Current emphasis has been placed on the evaluation of a two-zone process as a means of avoiding the factors that tend to produce conditions of poor fluidization. In this scheme a mixture of oxygen and nitrogen is introduced into the lower reaction zone (oxidation zone) formed by the uranium dioxide pellet bed, and fluorine is introduced into the upper zone (fluorination zone) which consists of a fluidized bed of alumina grain. The alumina in both zones is fluidized by the mixture of oxygen and nitrogen. Uranium oxide fines (U_3O_8) are formed in the lower zone and transported into the upper zone where they are removed by fluorination. (A schematic drawing of the two-zone fluid-bed reactor appeared in a previous Review.[8])

Two tests have been successfully completed[2] in the two-zone reactor, using a lower reaction temperature in the oxidation zone than in the fluorination zone. The bottom of the reactor was cooled so as to achieve a temperature of 140°C at the bottom of the oxidation zone and a temperature of 400°C at the top of the oxidation zone. Heat was applied to the reactor wall to maintain the upper fluidized bed at 500°C or above for fluorination. Conducting the oxidation at a low temperature allowed a close matching of the oxidation and fluorination rates. Both tests used a 12-in.-deep pellet bed charge of 8.8 kg of UO_2 and an amount of alumina grain sufficient to form a 24-in. upper zone under static conditions. Total processing time for complete fluorination in each test was about 17 hr. Overall fluorine efficiencies were about 50% with once-through gas flow. However, an efficiency of nearly 80% was obtained in one 5.7-hr period; and it is considered likely that similar or higher efficiencies can be obtained with closer fluorination control and with gas recycle.

Direct Fluorination of Plutonium-Uranium Oxide Mixtures. The direct fluorination process depends primarily on the conversion of the oxides of uranium and plutonium to hexafluorides in a bed of fluidized inert solids. Laboratory studies[2] have been concerned with the development of a fluorination scheme whereby optimum removals of uranium and plutonium from the inert solids can be achieved. This phase of the study is economically important since the inert solids will be discarded as waste. The laboratory tests have been carried out using static beds of uranium-plutonium oxide solid solutions mixed with inert solids which simulate the conditions likely to be encountered in plant processing. From the data obtained on the fluorination of the mixtures, several observations can be made regarding the optimum conditions for the removal of plutonium. Of the materials tested, Alundum (refractory grain alumina) has been found to be best suited for use as the inert solid, since its use results in the lowest retention of plutonium in the solid residue. For the initial period of fluorination, in which most of the uranium is removed from the solid mixture, a low temperature (450°C) and a low concentration of fluorine (10 vol.%) are desirable. In order to obtain satisfactory removals of plutonium in the last portion of the fluorination, it is necessary to use a higher temperature (550°C) and a higher fluorine content in the reacting gas (75 vol.%). A two-step procedure involving an initial oxidation of the oxide solid solution followed by two fluorination periods, one at 450°C and the other at 500°C, has resulted in plutonium removals of 99% or greater.

An engineering-scale high-alpha facility is being constructed[9] at ANL to study steps in the processing of spent reactor fuel by the fluoride-volatility process. The facility includes two alpha type boxes as shown in Fig. III-1. The larger box, 17½ ft high, 26½ ft wide, and 3½ ft deep, will hold all the plutonium-processing equipment. The smaller box, 10½ by 12¼ by 3½ ft, will contain the gas scrubber and process-gas metering manifold. A novel feature of these alpha type boxes is the location of bagout ports at the top of the boxes for removal of engineering-scale equipment. Materials may be added or removed from the boxes through 8-, 22-, or 30-in.-diameter openings using a bag-sealing technique. The bags, which are of polyvinyl chloride, are sealed by means of a dielectric

sealer. A 4-in.-diameter sphincter opening is available for introducing materials of small size by placing the materials in a standard-sized cylindrical cardboard or metal carton and pushing the carton through the sphincter opening. An empty carton is left in place to maintain the closure of the box.

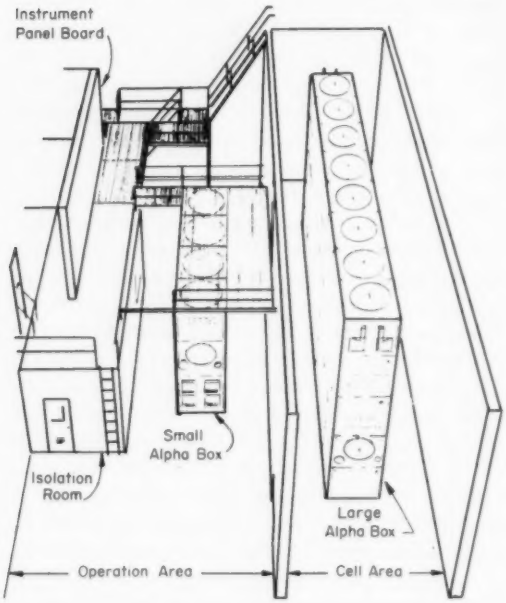

Fig. III-1 High-alpha engineering facility.[9]

The interior of the box is kept at a negative pressure of about ½ in. of water. The smaller box, which will be relatively uncontaminated, is ventilated with 100 cu ft of air per minute from the room; the larger box requires 400 cu ft of cell air per minute. In the large box the process off-gas, which consists mostly of oxygen, nitrogen, or inert gases, is mixed with the ventilation air in this box and is then passed through a spray scrubber and three sets of absolute filters in series. This scrubbed air together with ventilation air from the small glove box and cell ventilation air is then passed through another spray scrubber and absolute filter before being exhausted to the building environs.

Preparation of Fuels for Volatility Processing. Schmets and associates at CEN[6,7] (Mol, Belgium) are developing a fluorination process for recovering uranium from oxide and carbide fuels. To minimize fluorine requirements, it is necessary to convert the fuel to uranium tetrafluoride prior to fluorination. Uranium monocarbide may be converted directly to uranium tetrafluoride by reaction with either hydrogen fluoride[8] or sulfur tetrafluoride.[7] The reaction with sulfur tetrafluoride is rapid above 200°C; however, the uranium tetrafluoride product contains approximately 75% of the original carbon. In the processing of ceramic uranium dioxide fuel, the process sequence $UO_2 \rightarrow U_3O_8 \rightarrow UO_2 \rightarrow UF_4 \rightarrow UF_6$ is being studied. Each of these steps is being evaluated in demonstration tests in fluid-bed reactors. The reactions are carried out in the presence of aluminum oxide as an inert fluid-bed material.

An alternative method for processing power-reactor fuels is being studied at CEN.[6,7] This method involves the oxidation of the fuel, or an intermediate compound, to uranium trioxide prior to fluorination to uranium hexafluoride with sulfur tetrafluoride. Several tests have been made[7] on the fixed-bed oxidation of sintered uranium dioxide pellets by nitric acid vapors. It was observed that the reaction proceeds by the formation of UO_3 which continuously falls away from the pellet leaving a fresh exposed surface. Both $UO_3 \cdot 0.5H_2O$ and $UO_3 \cdot 0.8H_2O$ have been identified in the UO_3 product. The water can be removed from the trioxide by heating the product in dry air at 370°C.

The reaction of nitric acid vapors with U_3O_8 proceeded more rapidly than with sintered uranium dioxide. This suggests that, in the processing of uranium dioxide fuels, it would be advisable to incorporate a prior conversion of the uranium dioxide to U_3O_8 before oxidation to UO_3.

Corrosion. The resistance of nickel to attack by fluorine is attributed to the formation of an adherent scale of nickel fluoride on the nickel surface. Jarry et al.[10] have studied the mechanism of the nickel-fluorine reaction with respect to determining which species, metal or nonmetal, is the principal migrant through the nickel fluoride scale. The results of the study indicated that fluorine, rather than nickel, migrates through the fluoride scale during the course of the reaction. Fluorine could reach the metal surface by gross movement through pores and cracks in the fluoride scale or by some transport process through the bulk of the scale. Since the kinetics of the reaction have

been shown to follow a parabolic rate, it is most probable that the mechanism involves the transport of fluorine through the bulk of the nickel fluoride scale.[11]

Dixmier, Vincent, and Hasson[12,13] have reported the results of an investigation on the corrosion of iron and steel by uranium hexafluoride. Corrosion tests made with commercially pure iron[12] indicated that the mechanism of corrosion by uranium hexafluoride was complex and proceeded by several steps. In the first step, uranium hexafluoride reacts with the metal to give ferric fluoride and a lower fluoride of uranium (probably uranium tetrafluoride). In the second step, uranium tetrafluoride reacts with uranium hexafluoride to give an intermediate fluoride, the composition of which is determined by the process conditions. At low temperatures (approximately 100°C), the uranium and iron fluorides in the base film appear to be associated as complex fluorides. At higher temperatures (250°C), the complex does not occur; instead, individual layers of fluorides are formed—the intermediate fluoride of uranium being situated at the gas-solid interface and ferric fluoride adjacent to the metal. Only ferric fluoride is formed on the metal surface at elevated temperatures (greater than 250°C). In spite of precautions taken to purify the uranium hexafluoride employed in these tests, it was not possible to remove completely all traces of hydrogen fluoride. Therefore these observations may reflect slightly the action of hydrogen fluoride.

The corrosion of steel by uranium hexafluoride is greatly affected by the carbon content of the steel.[13] It was observed that graphite in steel undergoes rapid reaction, accompanied by destruction of the specimen through formation of interstitial compounds.

Fused-Salt Volatility Process

Design studies and cost estimates were prepared by ORNL for two on-site fluoride-volatility processing plants.[14] Each plant was assumed to be processing continuously irradiated LiF-BeF_2-ThF_4-UF_4 fuel from a one-region molten-salt converter reactor (MSCR) capable of producing 1000 Mw(e) [about 2500 Mw(t)]. One plant processed fuel at a rate of 1.2 cu ft/day, the second at 12 cu ft/day. A process flow sheet for the 12 cu ft/day plant (with Pa^{233} recovery) is shown in Fig. III-2.

In this study all the process operations were considered in enough detail for preliminary designs of vessels and equipment; complex vessels were considered more carefully to permit more reliable cost estimation. The process building was laid out for convenience of process operations and maintenance and was patterned after designs of other remotely operated plants that are the products of several years' experience and study. Cognizance was taken of the fact that the reactor and chemical plant are an integral operation and can share certain service facilities.

The chemical processing scheme consisted of volatilizing uranium as uranium hexafluoride by treating the molten salt with elemental fluorine at about 550°C. The hexafluoride was then collected by absorption on sodium fluoride and condensation in cold traps, reduced to uranium tetrafluoride in a hydrogen-fluorine flame, dissolved in makeup salt, and recycled to the reactor. Makeup fuel was supplied by purchasing fully enriched U^{235}. The lithium, beryllium, and thorium components of the fuel were discarded with the fission-product waste.

For the smaller plant the design study and cost estimates were based on two processing conditions: (1) retention of the waste salt for Pa^{233} decay and recovery by a second fluorination step and (2) discard of all Pa^{233} as waste after the first fluorination step. For the larger plant, only the case of Pa^{233} decay and recovery was considered. Cost estimates of the capital and direct operating charges are summarized as follows:

	Capital cost	Annual operating cost
1.2 cu ft/day plant with Pa^{233} recovery	$12,556,000	$1,103,000
12 cu ft/day plant with Pa^{233} recovery	25,750,000	2,241,000
1.2 cu ft/day plant with Pa^{233} discard	10,188,000	

The estimated capital costs for the two sizes of plants indicate that capital costs vary with the 0.3 power of the capacity rather than the 0.6 power usually associated with a capital cost vs. capacity curve for a chemical plant. The lower value for the slope suggests that more favorable processing economics will be realized with large processing plants.

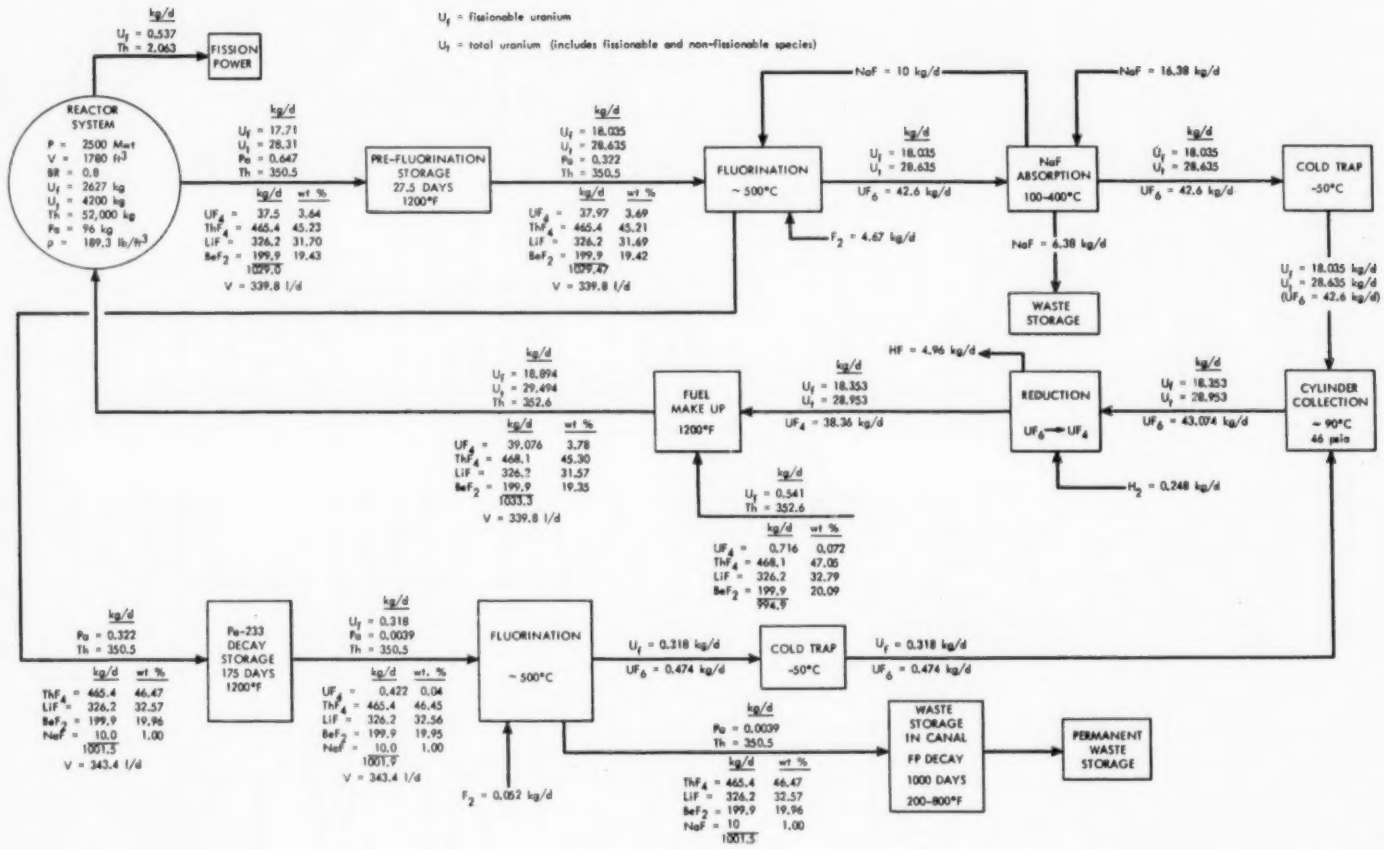
U_f = fissionable uranium
U_t = total uranium (includes fissionable and non-fissionable species)
FISSION POWER
REACTOR SYSTEM
PRE-FLUORINATION STORAGE 27.5 DAYS 1200°F
FLUORINATION ~ 500°C
NaF ABSORPTION 100-400°C
COLD TRAP -50°C
WASTE STORAGE
CYLINDER COLLECTION ~ 90°C 46 psia
REDUCTION
FUEL MAKE UP 1200°F
Pa-233 DECAY STORAGE 175 DAYS 1200°F
FLUORINATION ~ 500°C
COLD TRAP -50°C
WASTE STORAGE IN CANAL FP DECAY 1000 DAYS 200-800°F
PERMANENT WASTE STORAGE

Fig. III-2 Molten-Salt Converter Reactor. Process flow sheet for a 12 cu ft/day fluoride-volatility plant.[14]

The direct operating costs include labor charges corresponding to 104 employees for the 1.2 cu ft/day plant and 133 for the 12 cu ft/day plant. It is of interest to note the relation between operating and capital costs for each of the plants. When the operating cost is divided by the corresponding capital investment, the operating charge rate becomes 8.77 and 8.61%/year for the 1.2 and 12 cu ft/day capacities, respectively. These charges may be compared with a value of 15%/year that has been found to be generally applicable in the chemical industry.

The treatment of protactinium in this study was made to determine if there were sufficient value in the protactinium to justify its recovery from the waste. The capital cost of the 1.2 cu ft/day plant was estimated for the cases of complete Pa^{233} discard and for Pa^{233} retention until the undecayed Pa^{233} amounted to only 0.1% of the bred uranium. The economics favored complete Pa^{233} discard since considerable process equipment and building space were required for this "dead" storage. A more complete evaluation of the process might reveal that more favorable economics result from a nominal extension of the prefluorination storage period allowing a longer Pa^{233} decay period at this point. Increased process equipment, building, and inventory charges would have to be compared with the value of additional recovery of Pa^{233}. This latter analysis was not made in this study.

In the analysis of the 1.2 cu ft day plant employing Pa^{233} discard, the on-site, interim waste-storage time was optimized. The optimization was carried out by considering on-site storage costs vs. salt-mine permanent storage costs as a function of the age of the waste salt. The lowest total storage cost appeared to occur for an on-site holdup of about 1100 days before shipping to permanent storage.

Pyrometallurgical Processes

Pyrometallurgical processes for recovery of discharged reactor fuel materials include some processes in which metallic fuels are purified directly as metals and others in which metallic fuels and ceramic fuels are purified in liquid-metal--salt solvent systems. The potential economic advantages of these methods lie in their ability to handle short-cooled fuels, which permits minimum fuel inventories, and in the avoidance or simplification of chemical conversions. Pyrometallurgical, fluoride volatility, and aqueous processes were recently discussed in an article by Cairns.[15]

EBR-II Fuel-Cycle Facility

The Fuel-Cycle Facility has been built adjacent to the Experimental Breeder Reactor No. 2 (EBR-II) at the National Reactor Testing Station (NRTS) in Idaho to recover discharged EBR-II fuel material, initially by a process known as melt refining (a melting process in which most fission products are removed from molten uranium at 1400°C by vaporization and by selective oxidation reactions). Equipment for this facility is now being installed for receiving the first discharged fuel material, which is expected in mid-1964.

The major steps in the fuel-processing cycle are (1) disassembly of a reactor fuel subassembly to separate fuel elements (uranium pins sheathed in stainless steel), (2) decanning and chopping of uranium pins, (3) melt refining, (4) re-forming of uranium pins by injection-casting molten fuel into precision-bore Vycor molds, and (5) refabrication of fuel elements and fuel subassemblies. Two melt-refining furnaces and two injection-casting furnaces have been installed and tested.[16–18] The pin-processing machine shown in Fig. III-3 is used after injection casting to demold the cast fuel pins, shear them to length, reject short pins, weigh acceptable pins, measure their diameters, and detect flaws.[16] Two of these machines are being installed in the Fuel-Cycle Facility. The installation of numerous auxiliary equipment items is complete or in progress.[16,17,19]

Nitridation of Uranium-Fissium Alloy

In the Fuel-Cycle Facility, all operations in which the fuel is exposed to the atmosphere are conducted within an argon atmosphere in the so-called Argon Cell. The water and oxygen contents of this atmosphere are maintained at low concentrations, 5 and 20 ppm, respectively, but the nitrogen concentration can be several percent (up to 5%), depending on the inleakage rate and the rate of addition of high-purity argon. The consequences of this relatively high nitrogen concentration (i.e., the rate of nitridation of fuel in various geometries and temperatures and the effects on fuel processing) have been discussed from time to time in these

Reviews. A summary report of the experimental work on this problem has been issued.[20] The extent of nitridation of unirradiated fuel at three temperatures, as calculated from experimentally determined nitridation rates, is shown in Table III-2. One of the most important consequences of fuel nitridation is its effect on melt-refining yields. In a separate set of experiments, melt-refining yields were unaffected by 2 hr of exposure of unirradiated uranium-fissium pins at 650°C to a 5% nitrogen-argon atmosphere.

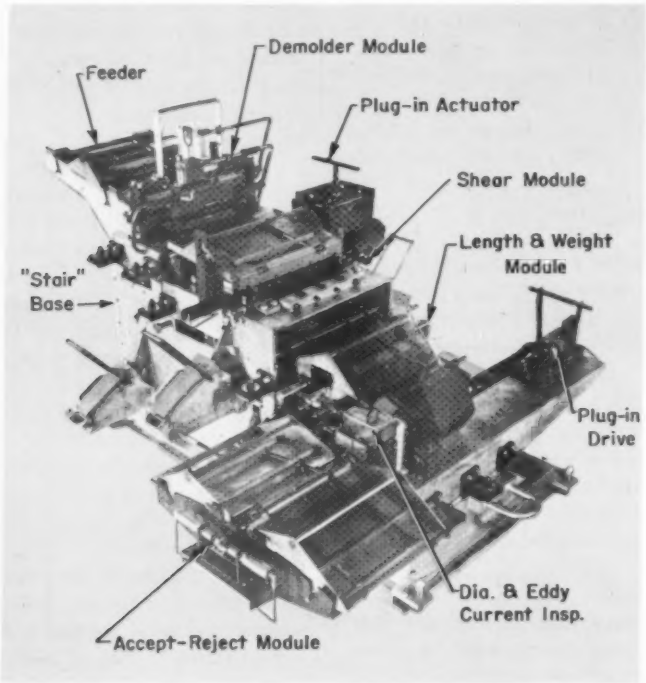

Fig. III-3 Pin-processing machine.[16]

Behavior of Iodine

Although major development work on the melt-refining process has been completed, work is continuing on problems related to the process. During melt refining, a Fiberfrax* fume trap (essentially an inverted porous cup placed on top of the melt-refining crucible) serves to condense or trap the condensable volatiles and particulate material. One of the materials condensed in the trap is fission-product iodine. The rate of desorption of iodine from the trap became of interest because of possible resulting problems in disposal of the trap.[21] With a temperature gradient across a 1-in.-thick section of Fiberfrax of 680 to 315°C (to simulate that expected in plant operation), the rate of iodine release was 0.16% per hour over the test period of 30 hr. This rate of release is sufficiently low that the trap may be canned for disposal without immoderate haste. The behavior of iodine was consistent with other indications that the condensed iodine activity is either cesium iodine or a material containing both cesium and iodine.

The general subject of iodine behavior in gas streams is discussed in two recent reports.[22,23] Although the subject is approached mainly from the standpoint of the behavior of iodine released from irradiated fuel, the information also has

* A product of The Carborundum Company prepared from amorphous fibers and a colloidal silica binder. The composition of Fiberfrax (wt.%) is: Al_2O_3 (51.2), SiO_2 (47.4), B_2O_3 (0.7), and Na_2O (0.7).

Table III-2 CALCULATED NITRIDATION OF UNIRRADIATED URANIUM–5 WT.% FISSIUM PINS AND INGOTS IN ARGON–5% NITROGEN ATMOSPHERE[20]

	197°C	308°C	504°C
K, $(g/cm^2)/min \times 10^{11}$			
Uncoated pins	0.236	5.60	186
Sodium-coated pins	0.958	15.2	325
Grams of nitrogen reacted per square centimeter in 100 min $\times 10^5$			
Uncoated pins	1.53	7.5	43
Sodium-coated pins	3.06	15.3	57
Nitride thickness in 100 min, mils			
Uncoated pins	0.008	0.039	0.224
Sodium-coated pins	0.016	0.099	0.296
Percent of total uranium nitrided in 100 min			
Uncoated pins (0.144 in. in diameter)	0.011	0.053	0.304
Sodium-coated pins	0.021	0.108	0.403
6-kg ingots (square)	0.002	0.011	0.040

much relevance to processing situations. In the first of these reports,[22] Atkins and Eggleton discuss the reaction of trace organic constituents in air with I^{131} released into air as carrier-free tracer or from irradiated uranium. Evidence was obtained for the formation of two classes of compounds. Methyl iodide was the major component of the first class. Some higher alkyl halides were also present. The nature of the compounds in the second class was not identified.

The second report[23] is an extensive treatise by Chamberlain of the physical chemistry of iodine and its removal from gas streams. The conclusions of this report are reproduced below:

(1) Fission product iodine released into a reactor enclosure or building will adsorb rapidly on to surfaces. Metallic surfaces adsorb more iodine and adsorb it more rapidly than other types of surface, but some adsorption occurs almost everywhere. The adsorption occurs at temperatures above the boiling point of iodine. It is partly reversible and partly irreversible. Some data on the rate of adsorption in various conditions is available, which enables estimates to be made of the likely hold-up of iodine in the containment.

(2) Adsorption on to dust and fume particles also occurs, and iodine which has undergone such adsorption is no longer readily available for adsorption on surfaces.

(3) Gas phase reaction of iodine with other trace constituents of industrial atmospheres also occurs.

(4) As a result of the phenomena (2) and (3), the iodine remaining in the gas phase after some time has elapsed from the initial release is likely to be in several physical and chemical forms. Methods of scrubbing iodine which rely on the chemical reactivity of elemental iodine (for example, scrubbing by caustic soda) have limited efficiency in this case.

(5) Activated charcoal is found to be a good absorbent for iodine in the gaseous forms so far encountered, at temperatures up to 200°C. When used in conjunction with an efficient mechanical filter (placed upstream of it), a system which is efficient in practice is obtained.

Purification of Zirconium and Beryllium

Claudson has studied the purification of zirconium by solid-state electrolysis and zone refining.[24] Metallic impurities (iron, nickel, copper, and aluminum) were reduced to a low level in the bulk metal by zone refining. Oxygen was concentrated at the anode by solid-state electrolysis with an effective diffusivity of 4.47×10^{-5} cm^2/sec per volt. Massive elimination of oxygen and nitrogen from zirconium has been achieved by the French by electron-beam melting.[25]

A patent has been issued to Love on the purification of beryllium by melting it with a rare-earth metal.[26] Impurities react with rare earths and settle to the bottom of the melt, leaving purified beryllium in the upper portion of the melt.

Development of Liquid-Metal and Salt Processes

Several processes are under development for the purification of reactor fuel materials in liquid-metal and salt systems. Purification of the fuel material is effected by selective extractions between immiscible metal and salt phases and by reactions within a single phase.

EBR-II Processes

Liquid magnesium-zinc solutions and liquid chloride salt systems are employed in skull reclamation and blanket processes that are under development at ANL for use in the EBR-II fuel cycle. The skull reclamation process complements the melt-refining process by providing for recovery and purification of fissionable material contained in melt-refining crucible residues. The blanket process serves to isolate the plutonium bred into the natural-uranium blanket of the EBR-II reactor in sufficient concentration (with respect to uranium concentration) for use in a reactor core. The flow sheets for both processes were given in a previous Review.[27] Recent work on the skull reclamation process has been mainly in the areas of process scale-up and resolution of minor process problems (Refs. 16, 17, 19, and 21).

Although separation of either the uranium or the plutonium from fission-product elements is not sought in the blanket process, a process demonstration run was recently made with lightly irradiated uranium to obtain information on fission-product behavior.[19,21] As expected, ruthenium (and probably also molybdenum) accompanied uranium through the process, whereas the rare earths, yttrium, and barium remained with the plutonium. Zirconium and tellurium activities were not accounted for and are believed to have been sorbed on solid surfaces. Although it is believed that only rarely would short-cooled blanket material be processed, the behavior of iodine was nevertheless investigated. The bulk of the iodine was gone from the magnesium-zinc process solutions at the end of the run, and the iodine did not appear in significant quantity in the furnace off-gases (about 0.1% of the initial total). It is believed that drossing reactions (e.g., formation of MgI_2) occurred and that easily condensable compounds volatilized and deposited on the upper walls and top flange of the furnace chamber.

Because of the highly corrosive nature of the zinc-magnesium processing solutions employed in the above processes, possible container materials are limited to tungsten and its alloys and to refractory oxides such as beryllia and alumina. In recent corrosion tests, tungsten and a 30% molybdenum-tungsten alloy were only slightly attacked (tungsten, 2 to 4 mils; the alloy, 2 to 8 mils) in a 500-hr exposure to a zinc-halide flux system at 1000°C, which is 200°C higher than the maximum process temperature.[21] From a corrosion standpoint, these materials are highly satisfactory.

The stability of solutions of uranium in magnesium-zinc alloys in the presence of impurities is important processing information. Uranium concentrations were not affected by additions of either beryllium or aluminum.[21] However, uranium was precipitated from solution by the addition of silicon,[21] an effect that had also been previously noted upon addition of carbon.

Mass Transfer of Cerium Between NaCl-KCl-$MgCl_2$ and Bismuth-Magnesium

In future processes it is anticipated that purification of fissionable materials may be effected by batch or continuous countercurrent

Table III-3 FUSED-SALT– LIQUID-METAL MASS-TRANSFER DATA[1]

Experiment No.	G_s, lb salt/(hr)(sq ft)	G_m, lb Bi/(hr)(sq ft)	C_{Mg}, ppm Mg in Bi	m, ppm Ce in salt/ppm Ce in Bi	$K_{os}a$, lb Ce/(hr)(cu ft)(lb Ce/lb salt)
Disk-and-Doughnut Column, 4 in. ID by 42 in. Long, with 5/8-in. Baffle Spacing					
2	1,610	78,800	16	215	200
3	2,220	21,000	8	600	9.6
4	10,040	24,800	76	20.9	1,240
5	1,840	22,800	94	15.2	606
7	2,220	47,900	76	20.9	676
8	2,100	87,700	112	11.7	467
11	9,300	22,200	155	7.2	1,110
14	1,960	63,400	149	7.6	716
Packed Column, 2.067 in. ID by 42 in. Long, Packed with 1/4-in. S.S. Spheres, 0.38 Void Fraction					
12	12,100	120,000	148	7.6	4,300
13	11,900	250,000	156	7.1	2,300

extraction operations between liquid-metal and liquid-salt phases. An 18-month study of the kinetics of liquid-salt–liquid-metal extractions at 500°C in a continuous countercurrent extraction column has been completed at Brookhaven.[1] The phases employed were a NaCl-KCl-$MgCl_2$ eutectic salt and bismuth containing a few parts per million of magnesium. The transferring solute was cerium. Most of the experiments were performed in a disk-and-doughnut column, but two were performed in a packed column. The volumetric mass-transfer coefficients, $K_{os}a$, based on the salt phase are summarized in Table III-3. These coefficients are not corrected for extraction outside the active section or for mixing in the direction of flow. However, these effects tend to cancel each other. Volumetric mass-transfer coefficients were appreciably higher in the packed column than in the disk-and-doughnut column.

On the assumption that the interfacial area per unit volume, a, is proportional to the mass velocity of the metal, G_m, lb/(hr)(sq ft), mass-transfer coefficients, K_{os}, were calculated using the relation: $K_{os} = (K_{os}a)/(1 \times 10^{-4}G_m)$. Although the results scatter somewhat (see Fig. III-4), they indicate that the mass-transfer coefficient is strongly dependent on the distribution coefficient.

Preparation of Metals by Reduction of Their Compounds

Liquid-metal and salt systems may be advantageous in the production of metals by reduction of their compounds. At ANL, oxides of thorium, plutonium, and uranium have been reduced by magnesium-zinc alloys from suspensions of the individual oxides in a molten halide flux.[28] Although the extent and rate of reduction of thorium oxide and uranium oxide are strongly dependent on flux composition (magnesium chloride must be present as a major constituent for complete reductions), the reduction of plutonium oxide appears to be relatively independent of flux composition (see Table III-4).[21,29] This difference in reduction behavior may represent a method for separating plutonium from thorium and uranium.

Two important pieces of information, which may be related to the mechanism of uranium oxide reduction from molten chloride systems, have been disclosed:

1. A U(V) species tentatively identified as UO_2^+ ion has been found in halide salt solutions and is formed by reactions of higher uranium oxides or oxychlorides with molten halide salts.[21]

2. A subhalide formed by slight dissolution of magnesium in magnesium chloride has been identified at Brookhaven by a chronopotentiometric method.[1] Preliminary indications are that the probable mechanism for the formation of the subhalide is $Mg^0 + Mg^{2+} \rightarrow Mg_2^{2+}$.

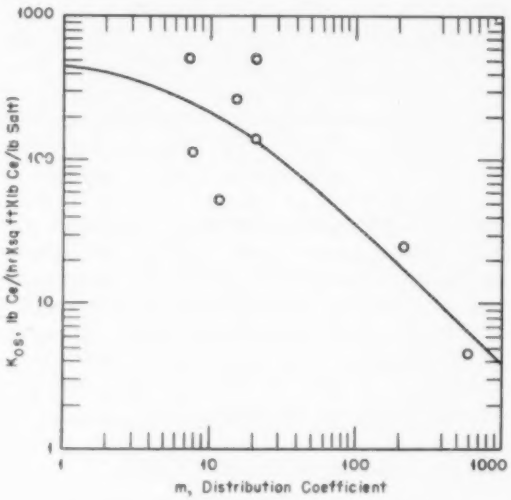

Fig. III-4 Estimates of the fused-salt–liquid-metal mass-transfer coefficients for the disk-and-doughnut column.[1]

Work on the direct reduction of uranium hexafluoride to uranium metal with sodium has been published by Scott.[30] This work, in which 146- and 584-g uranium buttons were prepared, showed the feasibility of continuous reduction of UF_6, with intermittent removal of uranium metal product. Up to 93.5% of the uranium content of UF_6 was recovered as uranium metal of acceptable purity. A remaining problem is the development of adequate construction materials for the reaction vessel.

Fundamental Studies Relative to Liquid-Metal Process Development

Knowledge of the chemistry of liquid-metal and salt systems is essential for the design of methods for processing nuclear fuels. Johnson and Chasanov at ANL are studying the thermodynamics of plutonium-zinc and plutonium-cadmium systems by a high-temperature galvanic cell method.[21] The activity coefficients of

Table III-4 EFFECT OF FLUX COMPOSITION ON REDUCTION OF PLUTONIUM DIOXIDE[21]

[Experimental Conditions: Temperature, 800°C; Mixing Rate, 800 rpm; Crucible, Tantalum; Atmosphere, Argon; Metal, 600 g Zinc–5 Wt.% Magnesium; Flux, 300 g (of Specified Composition); PuO_2, 7.13 g (1.0 Wt.% Plutonium in Metal Phase at 100% Reduction)]

Flux	Plutonium concentration at 120 min, wt.%		Reduction based on plutonium remaining in flux sample, %
	In metal	In flux	
95 wt.% $MgCl_2$–5.0 wt.% MgF_2	0.95	0.0109	99.5
47.5 wt.% LiCl–47.5 wt.% $CaCl_2$–5.0 wt.% MgF_2	0.88	0.0007	99.94
47.5 wt.% KCl–47.5 wt.% $CaCl_2$–5.0 wt.% MgF_2	0.97	0.0034	99.9
47.5 wt.% KCl–47.5 wt.% LiCl–5.0 wt.% MgF_2	0.95	0.0038	99.4

saturated solutions of plutonium in liquid zinc and cadmium have been measured over temperatures ranging from slightly above the melting point of the solvent metal to between 300 and 400°C above the melting point.[21] The solubility of plutonium in liquid zinc increases from 0.023 wt.% at 480°C to 3.27% at 725°C and may be represented by the equation:

$$\log (\text{at.\% plutonium in zinc}) = 8.612 - 10090T^{-1} + 1.461 \times 10^{6}T^{-2}$$

for the temperature interval[21] of 458 to 752°C.

The identification of binary phases in systems involving zinc or cadmium is the subject of a continuing study by Veleckis of Argonne. Recent additions to the fund of information in this area are identification of the intermediate phases that exist in systems of cadmium with cerium, praseodymium, and neodymium.[21] The zinc-vanadium phase diagram has been reported by Chasanov.[31] Nonstoichiometry in intermetallic compounds has been discussed by Elliott and Lemons of the Los Alamos Scientific Laboratory.[32]

Wiswall and Egan have reported on thermochemistry of bismuth solutions of thorium, protactinium, uranium, and plutonium and high-yield fission-product elements.[33] Data were obtained chiefly by galvanic cell measurements, but some thermodynamic information, particularly for fission-product elements, was obtained by equilibrations of two liquid-metal phases or liquid-metal and salt phases.

The following miscellaneous thermodynamic or physical-property information may also be of interest:

1. As measured by Bowersox and Leary, the solubility of carbon in plutonium increases[34] from 690 ppm at 700°C to 2350 ppm at 925°C.

2. Aldred and Pratt have measured the vapor pressure of liquid bismuth in the temperature range[35] 800 to 1050°K. Their results may be represented by the equation: $\log P$ (mm Hg) = $8.579 - (10{,}108/T)$.

3. Hesson and Burris found, by capillary-bath measurements, that the diffusivity of uranium in cadmium varied from 1.3×10^{-5} to 2.3×10^{-5} cm^2/sec over the temperature range[36] 450 to 650°C.

4. A progress report on the kinetics of reactions between liquid metals and liquid salts has been issued by King of the Massachusetts Institute of Technology.[37]

Construction Materials

Materials for Plutonium Systems

One of the major objectives of the LAMPRE-I reactor (Los Alamos Molten-Plutonium Reactor Experiment) is the evaluation of combinations of molten plutonium or plutonium fuel alloys and container materials in a reactor environment. In the July 1963 issue of *Reactor Fuel Processing*,[38] it was pointed out that intergranular penetration in weld areas had occurred in tantalum capsules containing a molten plutonium–10 at.% iron alloy. One capsule had failed. The intergranular penetration was attributed to oxygen contamination in the weld areas. In a continuing program of capsule examination, a large amount of intergranular and transgranular precipitates was found in the vapor-phase section of one capsule.[39] These

precipitates had the same morphology as tantalum nitride, and, because such precipitates were present to some extent in all capsules, it is suspected that reaction of tantalum with nitrogen in the inert cover gas is occurring. Gas analyses of the cover gas have shown that nitrogen builds up at a linear rate. It was calculated that this rate could be accounted for by diffusion of nitrogen through the stainless-steel reactor vessel walls.

Out-of-pile testing has shown that double-arc-melted tantalum is superior to electron-beam-melted tantalum for the containment of the plutonium-iron fuel.[39] Tantalum, tantalum–10 wt.% tungsten, and niobium were tested for 1000 hr for containing plutonium-cobalt-cerium fuels at 700°C. No failures of the tantalum-base capsules occurred, but some of the niobium capsules failed. With the alloys containing cobalt, formation of a Ta_6Co_7 layer on the tantalum has been observed.[39] Prior application of a tantalum carbide layer on the internal surface of the tantalum capsule results in a marked decrease or total absence of this reaction layer.

Besides the plutonium-iron and plutonium-cobalt-cerium systems, other alloy systems under study are plutonium-copper-cerium, plutonium-nickel-cerium, plutonium-magnesium-cerium, and uranium-manganese-plutonium.[39] Phase-diagram information and other properties of these systems have been reported or are under study at the Mound Laboratory.[40]

Materials for Mercury Systems

The corrosion resistance of materials, particularly carbon steels and refractory metals, to liquid mercury and mercury vapor at temperatures between 600 and 1400°F (316 to 760°C) is under study at Brookhaven because of the potential application of mercury as a turbine-driving fluid in reactor power plants. Twelve natural-convection boiling loops have been under test.[1] Titanium and zirconium have been employed as corrosion inhibitors (through formation of protective films), and magnesium has been used for wetting and deoxidation purposes. With and without an inhibitor present, the maximum penetration in various carbon steel and Croloy (2¼% chromium, 1% molybdenum) steel loops operated between 1100°F or 597°C (boiling and condensing) and 600°F or 316°C (deliberate cooling of condensate) was less than 1 mil in 5000 hr (Ref. 1).

The solubility of iron in mercury is pertinent to the corrosion of ferritic steels by mercury and has been measured at Brookhaven. The solubility decreased[1] from 1 ppm of iron at 700°C to 0.1 ppm at 450°C.

Mercury corrosion of metals other than ferritic steels is also of interest. A total of 5660 hr has been accumulated at Brookhaven in a loop fabricated of niobium–1% zirconium. In this loop, mercury is being boiled[1] at 1200°F (or 650°C). Argonne has reported that the presence of zirconium in mercury also reduces the attack on titanium (as reported above for ferritic steels). A similar reduction in corrosion of titanium was realized by the addition of 750 ppm nickel[19] or aluminum[17] to mercury. After 14 days of exposure in a nickel-saturated mercury solution at 538°C, duplicate titanium specimens had not suffered significant attack. A uniform single-phase protective layer 0.37 mil thick and composed principally of titanium and nickel had formed on each of the specimens.[19] With addition of 750 ppm aluminum, a powdery gray scum, presumably either a mercury-aluminum amalgam or aluminum oxide, formed on the titanium.[17] Addition of aluminum to a concentration of only 50 ppm accelerated the corrosion rate.[17]

Materials for Bismuth Systems

Japanese investigators have reported on the corrosion and erosion of ferritic steels by liquid bismuth containing magnesium and zirconium as corrosion inhibitors.[41]

Materials for Alkali Metal Systems

The corrosion resistance of refractory metals to sodium and its vapor between 1500 and 2400°F (815 and 1315°C) is under investigation at Brookhaven. Niobium–1% zirconium reflux capsules containing sodium boiling at 2200°F (1200°C) showed no detectable corrosion after 5000 hr of operation.[1] Forced-convection sodium loops fabricated of the niobium–1% zirconium alloy are now being put into operation. Materials for the containment of alkali metals are also being tested by the General Electric Missile and Space Division.[42] Capsule and thermal-convection-loop tests showed Haynes 25 alloy to have good corrosion resistance to potassium at 1850°F (1010°C). Problems of welding and component development are also discussed in the General Electric report.[42]

Materials for Thorium Systems

Various refractory oxide coatings on graphite crucibles have been investigated for melting thorium and thorium-uranium alloys.[43] The effectiveness of a coating was determined from the degree of crucible attack and the degree of carbon contamination. The quality of the cast material was also evaluated. Of the oxides investigated (Y_2O_3, ZrO_2, BeO, and ThO_2), best results were obtained with wash coatings of Y_2O_3 and ZrO_2 and with a flame-sprayed coating of ZrO_2.

Publications in Construction Materials Field

The Proceedings of a Conference on Corrosion of Reactor Materials Held in Salzburg, Austria, in June 1962, have been published.[44] Chick has issued a bibliography on corrosion of liquid metals[45] covering the period from 1957 to September 1962.

General Publications in Liquid-Metals Field

The first two issues of a bimonthly High-Temperature Liquid-Metal Technology Review have been published by BNL.[46] The reviews are designed to keep readers up to date on developments in liquid-metal technology, particularly as they relate to the space power program. For this reason, emphasis is not given to processing but, rather, to materials development and performance, heat transfer, fluid dynamics, component development, physical properties, and power systems.

Jackson and Mausteller have reviewed the chemical, physical, and nuclear properties of liquid metals having melting points equal to or less than that of aluminum.[47] Heat transfer, operational procedures, and equipment are among the subjects they discuss.

The twenty-first of a series of liquid-metal abstract bulletins has been issued by the MSA Research Corp.[48]

Other Nonaqueous Processes

Pyrochemical processes, which are being developed primarily for oxide and carbide reactor fuels, generally employ gases, molten salts, and liquid metals as reagents and process media. Pyrochemical methods are similar to pyrometallurgical methods in the use of high-temperature reactions, a capability for handling short-cooled fuels, simplicity, and compactness. The principal difference is that the major fuel constituents remain in the metallic state throughout a pyrometallurgical process. By aqueous solvent-extraction standards, both types of processes provide low fission-product decontamination, and they are regarded as most suitable for closed, remotely operated fuel cycles.

Oxide Fuel Processing

At Hanford, work has continued on the development of the salt-cycle process, a closed-cycle electrolytic procedure for recovering the uranium dioxide–plutonium dioxide fuel proposed for the Plutonium Recycle Test Reactor (PRTR).[49–52] The process involves mechanical decladding, oxidation of the fuel to a mixture of uranosic oxide and plutonium dioxide, and dissolution of this oxide mixture in a molten-chloride bath, e.g., LiCl-KCl, by sparging with chlorine or hydrogen chloride. In one version of the process, a portion of the uranyl chloride in the salt solution is reduced electrolytically with a graphite cathode, depositing plutonium-free uranium dioxide, which is a by-product of the process. The uranium and plutonium remaining in solution are then electrolytically codeposited as a mixture of the dioxides. Through appropriate control of the conditions, the uranium-to-plutonium ratio of this product can be controlled. In the other version of the process, the molten-chloride solution containing the dissolved uranium and plutonium oxides is sparged with an oxygen-chlorine mixture, which results in precipitation of plutonium dioxide. The uranium is then recovered by electrodeposition of uranium dioxide at a graphite cathode.

Further investigation of vibratory decladding and oxidation of uranium dioxide fuel pellets to uranosic oxide has been reported.[49] A total of 30 lb of uranium dioxide was removed from 10 one-half fuel-rod sections, oxidized to uranosic oxide, and dumped from the oxidizer. Losses of uranium oxide to the stainless-steel cladding were 0.072% for seven cold-swaged rods and 0.014% for vibratory-compacted rods. An average time of 2 min was required to declad each half rod.

Laboratory studies have continued on the electrodeposition flow sheet for the recovery of uranium dioxide–plutonium dioxide solid-solu-

tion fuels.[51] A deposit containing 23 wt.% plutonium dioxide has been obtained, in which the plutonium enrichment factor was 40 and the decontamination factor for promethium, a representative rare-earth fission product, was 6 (based on plutonium). This result was obtained with a platinum cathode at a current density of 0.05 amp/cm^2, a LiCl-KCl melt (mole ratio 1:1) at 575°C, and sparging with a 50% oxygen–50% chlorine mixture. It is thought that Pu(V) and Pu(VI) species formed by sparging with oxygen and chlorine are electrolytically co-reduced with the uranyl(VI) species, resulting in simultaneous deposition of uranium and plutonium dioxides. Several variables, such as current density, chloride ion activity in the melt, temperature, sparge gas composition, and cell geometry, control the relative rates of plutonium dioxide and uranium dioxide deposition, and therefore the plutonium enrichment. Since these variables interact with one another, and their useful ranges in some cases are limited by other considerations, careful choice and control of the conditions are required.[51] Several mixed uranium dioxide–plutonium dioxide samples have been prepared by this method for in-reactor fuel tests.

Further chemical studies have provided spectrophotometric evidence for the existence of plutonyl(VI) species in molten-chloride solvents.[49] Stable solutions having spectra corresponding to plutonyl(VI) chloride were prepared by sparging LiCl-CsCl solutions (mole ratio 1.4:1) containing 0.0103*M* plutonium with a 1:2 oxygen-chlorine mixture at 550°C. The solutions contained a minor amount of plutonyl(V) but no detectable Pu(III) or Pu(IV) species. Sparging a plutonyl(VI) solution with 100% oxygen at 550°C resulted in a precipitate, presumably plutonium dioxide. Sparging with 100% chlorine caused no spectral change in 20 min at 400°C, but at 550°C the Pu(V) and Pu(VI) concentrations decreased, and Pu(IV) appeared in the solution. Helium sparging of plutonyl(VI) solution at 400°C caused a rapid conversion of some Pu(VI) to the Pu(V) state, and subsequent sparging with hydrogen chloride resulted in conversion of the Pu(V) and Pu(VI) species to Pu(IV).[49]

Conversion of Pu(III) to the Pu(IV) state by saturating LiCl-CsCl solutions (mole ratio 1.4:1) with chlorine was also investigated. The reaction was at least 95% complete at 400°C, 75% at 550°C, and 40% at 700°C. The fact that these conversions are higher than those measured previously in LiCl-KCl solutions (mole ratio also 1.4:1) is consistent with results from studies with uranium, which indicated that the complexing power of molten-chloride solutions for the Pu(IV) species increases with the size of the solvent cation.

The effect of melt composition on the electroreduction of uranyl(VI) species has been examined through the use of chronopotentiometric measurements.[51] The diffusion coefficient for uranyl(VI) in NaCl-LiCl (mole ratio 1:1) at 704°C was about two-thirds of the value observed in NaCl-KCl (mole ratio 1:1) at 713°C. At 650°C in NaCl-LiCl, it was about three-fourths of the value in KCl-LiCl eutectic at the same temperature. The reaction appeared to be reversible and diffusion controlled at temperatures above about 550°C and at uranyl(VI) concentrations above about 6×10^{-3} molal.

Experiments have been reported in which cerium dioxide, used as a stand-in for plutonium dioxide, was precipitated from molten-chloride solutions in a gas-lift apparatus, using a chlorine-oxygen mixture as the gas.[49,50] The apparatus consisted of two concentric quartz tubes 50 mm and 10 mm in diameter, the 50-mm tube tapering down to a 20-mm opening at the bottom. The gas was admitted through the central 10-mm tube. In a LiCl-KCl melt (mole ratio 2.5:1) at 580°C, 24 hr of operation with a 70% oxygen–30% chlorine mixture resulted in precipitation of 85% of the cerium as the oxide.

In processing of PRTR fuel elements by the salt-cycle process after a relatively short cooling time of 15 days, it will be necessary to handle an off-gas stream laden with chlorine and fission-product I^{131} activity.[51] Since the use of a caustic scrubber to remove halogens would generate large amounts of radioactive waste solution, the possibility of liquefying the chlorine at about −100°C is being considered. This technique may permit efficient I^{131} removal, reuse of the chlorine, and small waste volumes. It was demonstrated that chlorine could be removed from a chlorine-air stream by a cold trap at −78°C and stored in a cylinder as a liquid.

Gaseous Low-Decontamination Process for Uranium Dioxide

Gaseous oxidation-reduction reactions are being utilized at Atomics International for low-decontamination processing of uranium dioxide reactor fuels. The remotely operated fuel cycle

involves pulverization of the refractory uranium dioxide pellets by alternate oxidation with air or dilute oxygen at 200 to 600°C and reduction with hydrogen at 600°C. The powdered uranium dioxide product is refabricated by cold pressing and sintering. The process does not produce significant fission-product removal, the objectives being to repair irradiation damage and to provide a means of reenriching the fuel. Multicycle processing and refabrication experiments on sintered uranium dioxide pellets reported earlier[53,54] have been published in a journal recently.[55]

Demonstrations of the process have been reported[56,57] in which 100-g quantities of uranium dioxide irradiated to burnups as great as 15,000 Mwd/metric ton were processed. A schematic flow sheet of the process is shown in Fig. III-5. The irradiated fuel was removed from the cladding by perforating the stainless-steel cladding and oxidizing the fuel with air or oxygen. The volume change occurring during oxidation caused the fuel to crumble and the cladding to split. Puncturing the cladding at close intervals along its length resulted in continuous longitudinal splitting, thereby avoiding plugs of uranosic oxide, which terminate the reaction by preventing further access of the gas. The decladding rate for high-burnup oxide did not differ significantly from that for low-level irradiated oxide, and the reaction rate did not change with temperature between 440 and 515°C. The decladding rate increased by a factor of 3 as the oxygen pressure was raised from 3 to 15 in. Hg abs. but was independent of further changes in oxygen pressure between 15 and 25 in. Hg abs. Puncturing the cladding at $1\frac{1}{2}$-in. intervals and tapping during decladding increased the instantaneous decladding rate to eight to ten times that for unpunctured tubes.

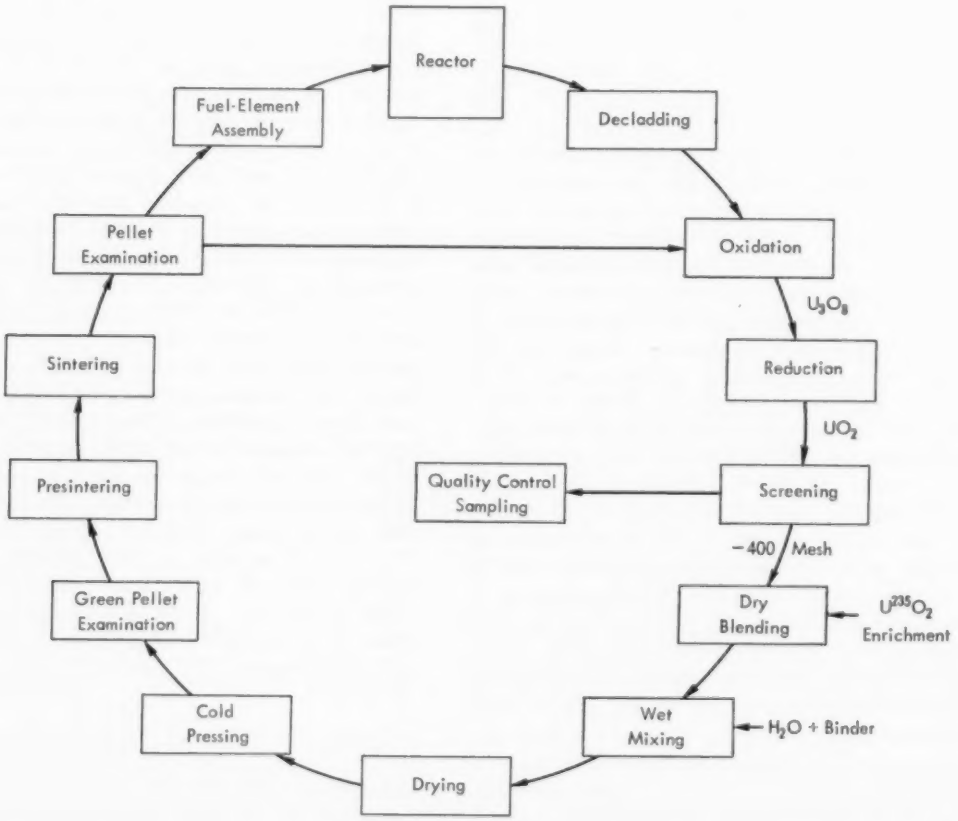

Fig. III-5 Basic flow sheet for oxidation-reduction processing[57] of UO_2.

It was shown that, if it becomes necessary to remove oxide plugs formed during decladding, this can be done by treatment with forming gas (15% hydrogen–85% nitrogen) at or above 515°C. Uranium losses to the cladding were less than 0.04% for fuel irradiated to 5700 Mwd/metric ton.

Since the uranium oxide powder produced by the decladding procedure described above is too coarse for uniform blending with enriched oxide and satisfactory fabrication by pressing and sintering, alternate oxidation-reduction was investigated as a method of particle-size reduction. Three or four such cycles (air oxidation at temperatures up to 600°C and reduction with forming gas at 600°C) were required to produce a powder with a tap density of 1.7 g/cm^3. Dusting of the powder appeared to pose no problems. The radioactivity detected in the off-gases included krypton, cesium, and ruthenium. Three oxidation-reduction cycles for fuel irradiated to 5700 Mwd/metric ton resulted in little or no removal of plutonium, cerium, cesium, rare earths, ruthenium, strontium, and zirconium-niobium. Irradiation level was not found to affect seriously the ability to achieve particle-size reduction, although about 2% of the highly irradiated powder could not be pulverized below 200 mesh, even after five cycles. Uranium losses did not exceed 0.1% in this step.

It was concluded that no equipment or processing problems had been encountered which would prevent scale-up of the process.

An economic comparison of a fuel cycle based on the Atomics International low-decontamination oxidation-reduction process (currently referred to as the AIROX process) and aqueous processing has been issued.[58] The study assumed a 1000-Mw(e) complex based on three thermal, uranium dioxide-fueled boiling-water reference reactors. The results indicated an economic advantage of at least 0.2 mill/kw-hr for the closed-cycle AIROX process over open-cycle aqueous processing. Although the U^{235} inventory and burnup requirements are greater for the AIROX process, the savings resulting from the simpler on-site closed fuel cycle were found to more than offset the additional burnup costs. The additional U^{235} burnup required to recycle a partial fission-product load for nine cycles (corresponding to 27 years) was about 15%. It is probable that, if this comparison were made for a fast-reactor system, greater savings could be achieved because the lower fission-product capture cross sections for fast neutrons would result in a lower U^{235} burnup penalty due to fission products. Specific arguments listed in favor of on-site closed-cycle processing were: (1) radioactive liquid waste handling and storage problems would be reduced, (2) no shipments of radioactive spent fuels over public routes would be necessary, and (3) the value of the transplutonium elements in the discharged fuel after a cumulative burnup of 135,000 Mwd/metric ton should be significant (e.g., it is estimated that 5 to 10 kg of Cm^{242} would be present in each reactor core).

Oxidation Behavior of Uranium Dioxide–Plutonium Dioxide Mixtures

Consideration of this type of processing for fast-breeder reactor fuels[58] leads to an interest in the oxidation behavior of uranium dioxide–plutonium dioxide mixtures. This subject has been investigated in a recent study at Harwell.[59] Solid solutions of the dioxides containing up to 80 mole % plutonium dioxide were oxidized in air at 750°C. From weight change measurements and X-ray powder crystallography, it was determined that different solid-solution oxidation products were formed, depending on the composition and physical state of the original oxide solution. The products were a cubic solid solution of the MO_{2+x} type containing 40 to 100 mole % plutonium dioxide, or an orthorhombic solid solution of the M_3O_{8-x} type containing 0 to 10 mole % plutonium dioxide. Either a single-phase product consisting of either one of these structures or a two-phase mixture of both was formed. There was no evidence for the formation of either pure plutonium dioxide or pure uranosic oxide.

A British patent on the low-decontamination oxidation-reduction process for uranium dioxide fuel has been issued to the U. S. Atomic Energy Commission.[60]

Carbide Fuel Processing

Further work on uranium monocarbide fuel processing has been reported by Atomics International.[61–63] Three processes are under study on a gram scale. In one, the carbide fuel is oxidized by oxygen, air, or carbon dioxide at temperatures of about 400 to 500°C; then the resulting oxide is reconverted to uranium monocarbide by carbothermic reduction at or above 1300°C. The second process involves conversion of the fuel to a mixture of uranium nitride and

carbon by treatment with nitrogen at about 900°C. This mixture is reconverted to uranium monocarbide by heating in vacuum above 1500°C. In the third process the uranium carbide fuel is anodically dissolved in a LiCl-KCl bath, depositing metallic uranium on a molybdenum cathode. This deposit is subsequently dissolved in mercury and converted to uranium monocarbide by distillation of the mercury in a propane atmosphere. Only the third process produces significant fission-product decontamination, the other two being merely refabrication procedures which permit reenrichment of the fuel and repair of irradiation damage.

Processing by the Carbide-Oxide-Carbide Cycle

It has been shown previously that the oxidation behavior of unirradiated uranium monocarbide is dependent on the history of the material, some being more "reactive" than others. Investigations of the oxidation of reactive uranium monocarbide by air and by oxygen at various pressures have been reported by Murbach.[64] It had been established earlier that freshly arc-melted uranium monocarbide was less susceptible to oxidation than material that had been exposed to laboratory air for a month or so. It was suspected that the latter material had become reactive as a result of a hydrolysis reaction with atmospheric moisture. Metallographic and X-ray examinations failed to reveal any differences in samples of the reactive and remelted material. However, a small weight increase (about 0.1%) accompanied the transition to the reactive state. Oxidation rates of the materials were measured by means of a thermobalance. The reactive uranium monocarbide ignited in oxygen at about 275 to 300°C and in air at about 350°C. The initial reaction appeared to form UO_{2+x} and continued heating produced uranosic oxide. The activation energy for the initial reaction in oxygen at 10 mm pressure was 7.1 kcal/mole.

Several batches of uranium carbide to serve as feed stock for vibratory-compaction tests have been prepared by the reaction of uranosic oxide-carbon agglomerates in vacuum, then heating to about 1800°C in hydrogen to remove the excess carbon.[61–63] Thus far, vibratory compaction of this material has resulted in lower densities than were obtained with crushed arc-melted carbide.

Scale-up studies have continued at Atomics International on the carbide-oxide cycle. In thermobalance studies of the conversion of uranosic oxide to uranium monocarbide by the use of carbon, a rapid weight loss was observed up to about 900°C (conversion of U_3O_8 to UO_2), a slow weight loss occurred between 900 and 1200°C, then the rate again became rapid above 1200°C (conversion of UO_2 to UC).[61] Further investigation of the 900 to 1200°C region indicated that the slow weight loss represented the beginning of uranium dioxide conversion to the carbide. The conversion of uranosic oxide to uranium dioxide was essentially complete at 900°C.

Processing by the Carbide-Nitride-Carbide Cycle

Freshly arc-melted uranium carbide has been shown to become more reactive with nitrogen after exposure to moisture. Work has continued on the behavior of reactive uranium monocarbide toward nitrogen. Since one possible explanation of the activation of uranium carbide is an increase in surface area, freshly melted uranium monocarbide (4.6 wt.% carbon) was ground under inert atmosphere, and reaction rates of various sieve fractions with nitrogen at 850°C were determined.[61] The reaction rate increased with decreasing particle size. In another series of experiments, Brunauer-Emmett-Teller (BET) measurements showed freshly cut arc-cast uranium monocarbide to have a surface area of less than 3 cm^2/g, whereas a sample exposed for 23 hr at room temperature to air saturated with water vapor had a surface area of 31 cm^2/g, and a sample exposed for 54 hr, 135 cm^2/g (Refs. 62 and 63). The surface area of the sample exposed 54 hr was in the same range as that observed previously for reactive carbide. In another study of the activation of uranium monocarbide, samples were exposed to varying water concentrations in water–isopropyl alcohol mixtures[63] at 70°C. The reaction rate of the treated carbide with nitrogen proved to be proportional to the square root of the water concentration.

It has been discovered that stoichiometric (4.80 wt.% carbon) uranium monocarbide which has become reactive can be deactivated by annealing under vacuum.[62,63] Based on reaction rates of the material with nitrogen, deactivation of the stoichiometric material was obtained at temperatures of 1070°C at 2×10^{-4} mm pressure, at about 1200°C at 1×10^{-3} mm, and at about 1400°C in 1 atm argon.[62] However, hyperstoichiometric uranium monocarbide (5.2 wt.%

carbon) showed little deactivation, even after heating 2 hr at 1515°C (Ref. 63), indicating that activation of the hyperstoichiometric material by exposure to water vapor is essentially irreversible. In the case of the stoichiometric material, a decrease in oxygen and hydrogen content (from 1530 to 800 ppm and from 22 to 4.5 ppm, respectively) was associated with the deactivation process.

Decladding of uranium monocarbide by reaction with nitrogen, the first step in the carbide-nitride-carbide process, has been investigated.[63] Uranium carbide rods, $\frac{1}{2}$ in. in diameter, were clad in stainless steel; then the cladding was pierced at 1-in. intervals. After exposure of the rods to air for varying periods of time in an effort to activate the clad carbide, the rods were exposed to nitrogen at temperatures between 700 and 850°C to determine the rates of fuel removal from the cladding. The method is considered to be promising.

Electrolytic Process for Processing Uranium Carbide Fuels

A report has been issued recently on the electrolytic process for processing uranium carbide fuels.[65] The three-step process is shown schematically in Fig. III-6. The uranium carbide fuel is dissolved anodically in a molten-halide electrolyte, forming a dendritic mass of uranium metal at the cathode. The cathode deposit, containing a substantial quantity of occluded salt, is immersed in mercury heated near its boiling point. The finely divided uranium and the mercury form a fluid quasi-amalgam phase that separates from the salt and other impurities. The amalgam is retorted under an atmosphere of hydrocarbon gas, forming uranium monocarbide and driving off the mercury. A final temperature somewhat below 600°C is needed for this step.

The LiCl-KCl eutectic was used as the electrolyte, since its low melting point (352°C) permitted later separation of the occulded salt as a liquid when the cathode was immersed in the near-boiling mercury. About 6 wt.% uranium tetrafluoride added to the salt produced satisfactory electrodeposition of metallic uranium. An argon atmosphere was used. Electrical contact with the dissolving uranium carbide anode was maintained by using a graphite crucible to hold the material. The anode reaction is

$$UC \rightarrow U^{3+} + C + 3e^{-}$$

A satisfactory cathode material must be inert to the salt bath, the deposited uranium must adhere to it, and it must be inert to hot mercury.

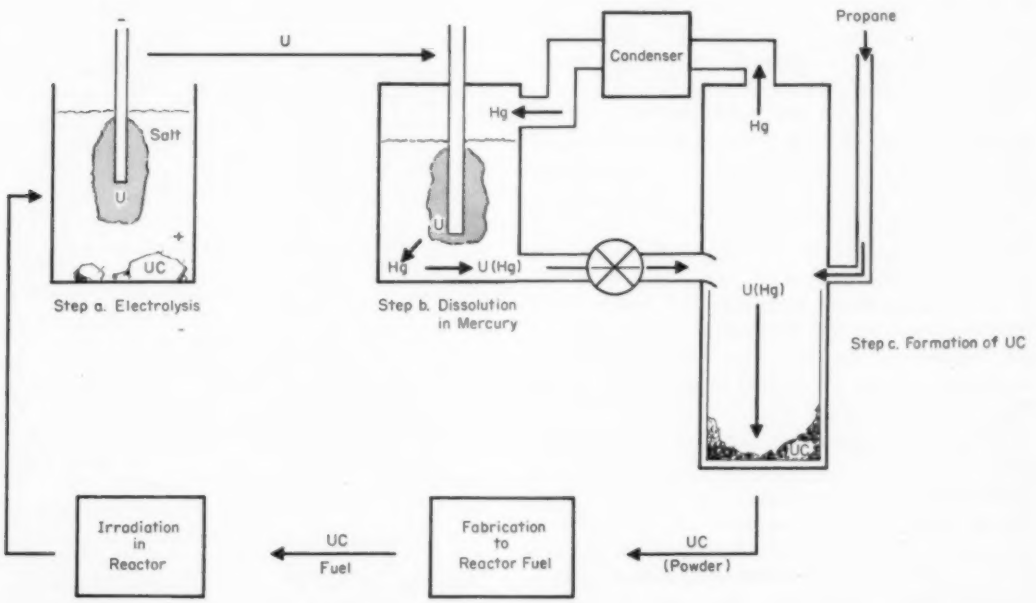

Fig. III-6 Flow sheet for processing of UC.[65]

Table III-5 OBSERVED DECONTAMINATION FACTORS* FOR UC ELECTROLYSIS[65]

Element	Deposit 1	2	3	4	5	6
Barium	2.1×10^3	2.0×10^3	3.3×10^3	5.8×10^3	2.0×10^3	4.2×10^3
Strontium	7×10^3	6.7×10^3	5.4×10^3		3.0×10^3	5.2×10^3
Rhenium	1.1×10^3	5.9×10^2	4.1×10^2	5.6×10^2	8.5×10^2	4.3×10^2
Cerium	85	65	53	58	86	51
Ruthenium†			6×10^2		8×10^2	1.1×10^3
Zirconium	1	1	1	3	2	3

* These factors (except for ruthenium) indicate decontamination observed in going from the salt-bath phase to uranium carbide powder, the final product. Overall decontamination factors at one throughput can be greater, but never less, than these values.
† Overall decontamination factors at one throughput.

Molybdenum was used as the cathode material, although several other metals are also considered suitable. In the final series of experiments, with a starting cathode area of 7.8 cm^2, 10 wt.% uranium tetrafluoride in the melt, and currents of 5 or 10 amp, the cathode efficiency was from 51 to 60%. It was found that the succeeding process step, i.e., dissolution of the cathode deposit in mercury and separation of the fluid quasi-amalgam phase from the molten salt, could be accomplished with little difficulty. During retorting of the quasi-amalgam in hydrocarbon gas, the following reaction occurs:

$$m\mathrm{UHg_4} + \mathrm{C}_m\mathrm{H}_n \rightarrow m\mathrm{UC} + 4m\ \mathrm{Hg} + \frac{n}{2}\mathrm{H_2}$$

Dried propane was used as the atmosphere in experiments with inactive material. X-ray diffraction data on the product showed no phases other than uranium monocarbide.

In addition to several experiments with inactive material, tracer-irradiated uranium carbide was processed by the cycle. Essentially the same procedure was followed, except natural gas was substituted for propane in the retorting step. Fission-product decontamination factors for the electrolysis step are listed in Table III-5. Because noble metals are removed at the anode, an overall figure for the process is given for ruthenium. In all cases, these should be regarded as the minimum values that can be expected from the process.

It was concluded that, although further development work is needed, the process is promising. The operations are simple, high fission-product removals are obtained for a process of this type, and the product appears to be good-quality powdered uranium monocarbide which may be amenable to direct fabrication.

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Section

IV

Reactor Fuel Processing

Waste Disposal

Liquid-Waste Treatment by Ion Exchange and Foam Separation

Ion Exchange

The treatment of large volumes of low-level-activity process wastes that originate from many sources in the processing of nuclear fuels is being investigated at Oak Ridge National Laboratory (ORNL).[1,2] A scavenging ion-exchange process is being developed for wastes of this type since their evaporation to solids or their storage as a liquid waste would be prohibitively expensive. This process employs a head-end treatment step which involves precipitation of a portion of the activity with sodium hydroxide, clarification of the water in a gently agitated sludge-column clarifier, and filtration of the effluent from the clarifier unit. The clarified and filtered effluent is then passed through a carboxylic-phenolic resin for sorption of Cs^{137} and Sr^{90}.

In laboratory tests, water clarification techniques continue to be studied in an effort to reduce the total hardness of the effluent from a clarifier unit.[2] The reduction of the hardness ions (magnesium and calcium) in the stream to the clarifier unit increases the quantity of solids removed by the sludge blanket and reduces the burden on the polishing filters. In the precipitation step of the process, the process plant waste is made 0.01*M* in NaOH and 5 ppm in ferrous ion (as ferrous sulfate). However, when this treatment was applied to ORNL low-level-activity plant waste, difficulty was encountered in reducing the total hardness of the effluent from the clarifier unit, which was attributed to the presence of hexametaphosphate, a constituent of some commercial detergents and decontaminating solutions.

The effect of the presence of phosphate on the residual hardness of the clarifier effluent stream was then studied in laboratory-scale clarifier tests using tap water. The amount of sodium carbonate required to reduce the effect of phosphate was also determined. The presence of 1, 2, 3, or 5 ppm phosphate (added as ortho-, hexameta-, or polyphosphates) in the tap water increased the hardness ions of the clarifier effluent stream from about 4 ppm (with no phosphate present) to as high as 70 ppm (as $CaCO_3$). The results indicate that phosphate ion will create hardness problems regardless of its ionic species. The addition of 0.005*M* Na_2CO_3 was sufficient to reduce the residual hardness content from about 42 ppm to less than 14 ppm when the amount of phosphate present was less than 3 ppm; however, 0.01*M* Na_2CO_3 was required to reduce the hardness content from 70 to 24 ppm (in 24 hr) when about 5 ppm phosphate was present.

ORNL has issued two topical reports that summarize the results of laboratory- and small-scale engineering tests[3] and pilot-plant demonstration runs[4] on the removal of activity from low-level-activity ORNL process wastes by this scavenging ion-exchange process. (This work has been covered in a previous issue of *Reactor Fuel Processing*.[1])

Decontamination of Purex Tank-Farm Condensate at Hanford

At Hanford, experiments were continued in the Micro Pilot Plant (MPP) to develop an ion-exchange process for the decontamination of Purex tank-farm condensate. In previously reported experiments[5] it was shown that both cation and anion exchange are necessary for adequate removal of ruthenium from steam-stripped

Purex tank-farm condensate. The relatively low cost of cation-exchange materials and their high capacity for ruthenium and other isotopes allow economic disposal of this material after being exhausted.[6] However, the strong-base anion-exchange resins are considerably more expensive and have limited capacity for ruthenium; thus they must be regenerated and reused. Although the steam-stripped condensate is very clear, a small amount of organic matter is present as a filterable particulate matter. The organic matter also has adhesive properties that cause considerable resin particle agglomeration. It is necessary to use mechanical means to break up the agglomerate and solvent treatment to dissolve the organic matter adhering to the resin beads before regeneration can be accomplished. Therefore a filtration step is considered necessary to protect ion-exchange beds that are to be regenerated.

Two ion-exchange runs[6] were made to investigate the use of a precoat type filter in which cation-exchange material of small particle size is used as a filter-aid material so that both filtration and cation-exchange adsorption could be done in a one-step unit operation. In these runs the decontamination factors of only strontium and cesium were studied. In the first run a 1.7-cm-thick precoat filter bed of a strong acid cation resin (Dowex 50W; 200 × 400 mesh) in the hydrogen form was evaluated. The steam-stripped condensate that was acidified with nitric acid to *p*H 3.2 was passed through the precoat bed at a flow rate of about 5 gal/(min)(sq ft). A decontamination factor for strontium of greater than 1000 was maintained for over 5000 column volumes. The decontamination factor for cesium varied from 700 at the beginning of the run to 70 at 2250 column volumes when its MPC_w value was reached. The pressure drop across the filter bed increased from 2.1 psi at the start of the run to 3.3 psi at the end of the run.

In the second run a 2.0-cm-thick precoat filter bed of acid-washed clinoptilolite (100 × 230 mesh) in the sodium form was evaluated. The steam-stripped condensate that was acidified with nitric acid to *p*H 4 was passed through the precoat filter bed at a flow rate of 5 gal/(min)(sq ft). The decontamination factors for strontium and cesium ranged from 100 to 400 during the run in which a total of 4600 column volumes of waste was processed. The concentrations of strontium and cesium in the effluent did not exceed their respective MPC_w values during the entire run. The pressure drop across the filter bed did not exceed 2 psi for the run.

Adsorption of Cesium on Clay Minerals

The U. S. Geological Survey[7] has issued a report which describes the progress that has been made in the study of the exchange adsorption of cesium from solution for the more common cations found in clay minerals. This exchange of ions in solution for those on natural ion exchangers plays an extremely important role in controlling the concentrations of radioactive substances in water and the rate of movement and dispersal of such substances in the environment.

The amount of cesium adsorbed by clay minerals (kaolinite, halloysite, montmorillonite, and "illite") was measured in solutions in which the concentration of cesium ranged from $10^{-1}N$ to approximately $10^{-10}N$ and in which either sodium, potassium, calcium, or magnesium ions were also present in various concentrations (from $2 \times 10^{-1}N$ to $2 \times 10^{-3}N$). These ions are considered the most abundant of the cations present in natural water. Their use also provided a means of studying the effect of size and valence of the cation on the exchange adsorption of cesium.

The distribution coefficients for the adsorption of cesium on clay minerals were calculated. The distribution coefficients were found to be independent of the cesium concentration at very low cesium concentrations (less than $10^{-6}N$) and for a constant concentration of the cation competing with cesium for adsorption sites. The logarithm of this limiting value of the distribution coefficient was found to be a linear function of the logarithm of concentration of the competing cation. This observation indicates that the distribution coefficient may be very useful for predicting the movement of cesium transported by water, particularly if similar results are obtained for more complex systems. At higher cesium concentrations (greater than $10^{-6}N$), the distribution coefficients were found to depend on both the cesium concentration and the nature and concentration of the competing cation. The results also indicated that cesium ions may be expected to move the shortest distances through soils and clays in which sodium is the predominant exchangeable cation.

Foam Separation

ORNL is continuing its investigation of the removal of Sr^{90} from ORNL low-level-activity plant wastes. In order to increase the efficiency of foam separation as a decontamination method for these wastes, it has been previously shown desirable to decrease the hardness content of waste streams by means of a pretreatment step.[8] This step consists of precipitation of the hardness ions (magnesium and calcium) by the addition of 0.005*M* each of sodium hydroxide and sodium carbonate, and coagulation with 2 to 5 ppm ferric ion (as ferric chloride), which also serves to reduce the interference effect of phosphates. These reactions are carried out in a single-chamber agitated-sludge bed column clarifier.

In laboratory-scale tests[2] that were performed in 3-, 4-, and 9-in.-diameter column clarifier units and at flow rates that ranged from 7.4 to 60 gal/(sq ft)(hr), the effect of column diameter, sludge height, and flow rates on the precipitation of calcium and magnesium from ORNL tap water and low-level-activity plant waste was studied. Within the range of column diameters and flow rates studied, the head-end precipitation step reduced the hardness content from 100 to 2 to 4 ppm (as $CaCO_3$). For equal flow rates the operation and physical behavior of the various columns (up to 9 in. in diameter) were very similar. Thus there would appear to be no unusual problems in the scale-up of this pretreatment process. The ratio of height of the sludge bed to the diameter of the column was not critical in the region from 4 to 0.5, which suggests that this ratio may be reduced still further.

The presence of phosphates in the waste and the occlusion of calcium carbonate particles by the magnesium and ferric hydroxides and organic matter in the waste tend to decrease the rate of hardness removal. The effect of the phosphate can be reduced by *p*H control and by the addition of sodium carbonate and ferric chloride. The occlusion of calcium carbonate particles can be minimized by gentle agitation that liberates the heavier calcium carbonate particles from the light magnesium and ferric hydroxide precipitates and the organic matter.

In five engineering-scale runs[2] that were carried out in a 6-in.-diameter column, a one-step foam-separation procedure was investigated in which the precipitated hardness ion solids (calcium carbonate and magnesium hydroxide) and strontium are concurrently removed. ORNL tap water containing added Sr^{89} [about 10,000 counts/(min)(cm^3)] was used to simulate low-level-activity ORNL plant waste. Dodecylbenzene sulfonate (DBS) was used as the surfactant. The concentration of the surfactant was 270 ppm, except for a portion of one run in which it was increased to 350 ppm. In all runs the hardness ions in the simulated ORNL plant waste were first precipitated, and the resultant slurry was then used as feed to the foam column.

In the first run, both 0.005*M* NaOH and 0.005*M* Na_2CO_3 were added to the simulated plant waste to precipitate the dissolved magnesium and calcium. The results of this run showed that it is possible to use foam for removing precipitated solids concurrently with the separation of strontium. The decontamination factors for strontium ranged from 81 to 162.

In the second run, 0.004*M* NaOH, 50 ppm Na_3PO_4, and 10 ppm $FeCl_3$ were added to the simulated plant waste to precipitate the hardness ions. The precipitate was in the form of large, wet flocs which seemed to cause increased channeling in the foam column. The decontamination factors for strontium were lower in this run than in the first run and were about 25 to 30.

In the remaining three runs, 10 ppm of a proprietary detergent (Fab) was added to the simulated plant waste. In each run the dissolved magnesium and calcium were precipitated by the addition of one of the following combinations of reagent solutions: (1) 0.005*M* NaOH – 0.005*M* Na_2CO_3; (2) 0.004*M* NaOH – 50 ppm Na_3PO_4; and (3) 0.005*M* NaOH – 0.005*M* Na_2CO_3 – 0.005*M* precipitated $CaCO_3$ as a seed slurry. In these runs the decontamination factors for strontium were low, about 2. The rate of precipitation of the hardness solids was low, and the precipitation was observed to continue to take place in the samples of the effluent from the foam column. For example, the addition of 0.005*M* NaOH and 0.005*M* Na_2CO_3 to a simulated plant waste containing 10 ppm Fab did not give any visible precipitation within 30 min; although, in the first run in which Fab was not used, visible precipitation occurred within 1 min after the addition of these same reagents.

Conversion of High-Level-Activity Wastes to Solids

Spray Calcination

At Hanford, radiant-heat spray calcination[9] as a method for the treatment of Purex-process high-level-activity aqueous wastes continues to be studied. In radiant-heat spray calcination, the feed solution is sprayed into the top of a heated column that is maintained at 700 to 750°C. Spray droplets are converted to solids, and some salt decomposition is achieved. The dried residue falls into a melt pot that is used to effect a reduction in volume of the residue. In the melt pot, which is maintained well above the melting temperature (typically 750°C) of the dried residue, further decomposition and melting of the dried residue take place continuously.

A run[6] was made with neutralized full-plant-level-activity Purex waste (1WW) in the radiant-heat spray calciner in order to determine the effect of high *p*H on the behavior of ruthenium. The results of previous laboratory studies[10] had indicated that there would be negligible volatilization of ruthenium from high-*p*H Purex wastes. The acidic Purex 1WW waste (composition shown in Table IV-1) was adjusted to *p*H 10 with sodium hydroxide. Sugar was then added to the alkaline waste to promote pyrolysis of sodium nitrate and to produce a meltable sodium carbonate matrix. The results of the run showed that the use of an alkaline feed significantly reduced the volatilization of ruthenium. The decontamination factor for ruthenium was similar to that of the other fission products (about 1×10^9). The cumulative decontamination factors for ruthenium, cerium, cesium, and zirconium-niobium which were achieved by the various components in the spray calciner and off-gas treatment installation are shown in Table IV-2. The contamination of the off-gas was probably caused by entrainment. The decontamination factors varied throughout the run but showed a generally increasing trend as the run progressed, presumably a result of not blowing back the stainless-steel filters until the end of the run. The decrease in ruthenium decontamination factor across the silica-gel bed absorber was most likely caused by the desorption of ruthenium left on the silica-gel bed from previous runs. The density of the calcine was 3.1 g/cm^3. The volume of the melt was about 1 liter, which is equivalent to a volume reduction of 5 based on the volume of the original acidic Purex waste.

Table IV-1 COMPOSITION OF FULL-PLANT-LEVEL-ACTIVITY PUREX WASTE (1WW)[6]

(Major Components)

Ion	Molarity
H^+	2.4
Fe^{3+}	0.29
Al^{3+}	0.11
Na^+	0.61
NO_3^-	3.7
SO_4^{2-}	0.24
Fission products	About 0.02*M*
Settled solids	About 12% of total volume
Radiation level	About 10^6 r/(hr)(liter) at 1 ft

The use of a draft tube was tested in the Hanford semiworks-scale radiant-heat spray calciner to determine its effectiveness in minimizing the deposition of calcine on the calciner walls. The function of the draft tube is to control the nature and direction of gas currents in the calciner. Without a draft tube, the eddy currents set up are largely responsible for the deposition of calcine on the calciner wall. This wall deposit reduces the heat flux from the furnace to the interior of the calciner, and the capacity of the calciner is thus reduced. The draft tube is a hollow cylinder that is unrestricted at the bottom, is concentrically placed and fastened to the reactor top, and contains inlet ports on the upper wall. In operation the gases that are drawn downward by the action of the atomizing feed nozzle are replaced by gases moving upward between the draft tube and the wall of the calciner. The calcine contained in this gas is dry and is much less likely to deposit on the calciner wall.

Two sizes of draft tubes (Mark I and Mark II) were tested in the spray calciner using simulated, nonradioactive Purex-process waste solutions [concentrated acid waste (1WW) and formaldehyde-treated waste (FTW)]. The semiworks-scale spray calciner is 10 ft in length and 18 in. in diameter and has a nominal capacity of 20 gal/hr.

Of the two draft tubes tested, the Mark II draft tube was more effective in minimizing wall deposits. It was 7 ft in length by $13\frac{3}{4}$ in. in diameter and had a ratio of the cross-

Table IV-2 FISSION-PRODUCT DECONTAMINATION OF SPRAY-CALCINER OFF-GAS[6]

Calciner component	Cumulative decontamination factors			
	Ru	Ce	Cs	Zr-Nb
Spray calciner and porous stainless-steel filters*	2×10^3	2×10^3	2×10^3	2×10^3
Condenser	1×10^5	3×10^3	1×10^5	5×10^5
Electrostatic bubble scrubber	1×10^5	3×10^5	1×10^5	5×10^5
Silica-gel bed	5×10^7	3×10^5	1×10^5	5×10^5
Cambridge absolute filter	2×10^5	5×10^5	1×10^5	1×10^5

*By analysis of the condensate.

sectional areas of the inside of the draft tube to the inside of the calciner of 0.58. Under most of the conditions tested, the only calcine deposit was on the calciner wall and was a 1- to 2-ft-wide band at the level of the draft-tube outlet (7 ft from the reactor top). With both simulated 1WW and FTW feed solutions, the amount of this deposit reached an equilibrium value, i.e., the calcine deposit flaked off at a rate equal to the deposition rate. This deposit could be easily removed by brushing or by a jet of water. It appears that lengthening of the draft tube may reduce this deposit.

It was estimated that heating of the feed stream to near boiling (prior to spraying) should decrease the energy requirement of the calciner by approximately 15%. Thus, for a given heat flux, the capacity of the calciner should be increased. This was verified in test runs in which the capacity of the calciner (with air atomization) was increased 40 to 50%. This additional capacity may have been the result of increased convective heat transfer at the higher recirculation rates around the draft tube at the higher feed rates and to changes in atomization characteristics. A comparison of the calciner capacity curves from runs made with and without a draft tube suggests increasing convective heat transfer with increasing rates with a draft tube.

In runs with the Mark II draft tube, the effects of furnace temperature and feed rate on the density of the calcine produced from a simulated FTW feed adjusted for calcine melting were determined. The runs were made at furnace temperatures between 650 and 800°C and at feed rates of 10 and 15 gal/hr. The results of the runs showed that increasing the feed rate and decreasing the furnace temperature both tend to lower the density of the calcine produced (Fig. IV-1).

Allemann and Johnson,[11] at Hanford, have summarized in a recent publication the results of the radiant-heat spray calcination of simulated, nonradioactive, waste solutions (Purex, TBP-25, Darex, and Zirflex) which was carried out in an 8-in.-diameter by 10-ft-long reactor column. The experimental equipment is described, and the effect of various operating variables on factors such as capacity and completeness of calcination are discussed. (This work has been covered in previous issues of *Reactor Fuel Processing*.[12])

Pot Calcination

At ORNL the pot-calcination process for converting high-activity-level wastes to solids is being studied on both a laboratory and engineering scale to provide design information for the

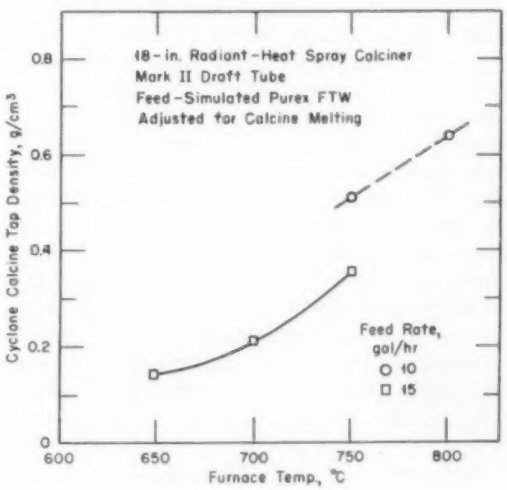

Fig. IV-1 Effects of furnace temperature and feed rate on calcine density.[6]

construction of a pilot plant to be installed at Hanford.[13] Development work has been with synthetic Purex, Darex, and TBP-25 wastes. The first phase of the program is concerned with direct calcination processes in which melting does not occur and little or no additives are combined with the wastes. In the second phase, enough additives are combined with the waste for subsequent formation of a glasslike material in which the fission products are fixed.

In two engineering-scale, semicontinuous experiments,[2] in which simulated TBP-25 waste (containing added Ru^{106}) was evaporated, calcined, and melted (at about 1050°C) to a glass, the effect of the addition of either phosphite or phosphate as a fluxing agent on the volatilization of ruthenium was investigated. In both runs the calciners were operated with a slowly rising liquid level. The simulated TBP waste was made 2.0*M* in NaH_2PO_4 and 0.25*M* in PbO for the first run and 2.0*M* in NaH_2PO_2 and 0.25*M* in PbO for the second run. The results of the two runs showed that the production of the glass was accompanied by volatilization of about 24% of the ruthenium regardless of whether phosphite or phosphate was added as the fluxing agent.

In laboratory-scale experiments on the volatilization of sulfur trioxide (SO_3) from glass melts,[2,14] simulated high-sulfate Purex waste oxides (from 39 to 43 wt.%) were incorporated into glass melts by fluxing with sodium hypophosphite (NaH_2PO_2) and varying combinations of other fluxing agents (phosphorous acid, sodium hydroxide, calcium hydroxide, magnesium oxide, and sodium perborate). All melt compositions investigated lost some sulfate (from 2 to 22%) when heated to 100°C above the softening point[2] (850 to 950°C). At these temperatures all the melts investigated were sufficiently fluid to pour easily. This temperature (100°C above the softening point) should, therefore, be considered the operational maximum for a practical fixation process. No true glasses were formed among the melts investigated. In general, SO_3 volatility was less for those melts that had a greater ratio of gram equivalents of cations (sodium, calcium, and magnesium) to gram equivalents of nonvolatile anions (sulfate, phosphite, and perborate).

Hanford Pot Calcination of Full-Plant-Level-Activity Purex 1WW Waste

At Hanford, two pot-calcination runs[6] were made with full-plant-level-activity Purex waste (1WW) in order to produce a calcine containing sodium nitrate for long-term stability observations (to determine the extent of radiolysis of the sodium nitrate) and to determine whether adequate off-gas decontamination could be obtained with less complex off-gas systems than have been used in previous runs.

In the first run, 26 liters of as-received 1WW waste (no glass-forming agent or modifiers added) were calcined. The resulting calcine did not melt since the as-received 1WW waste was not of a composition which would produce a melt. The weight of calcine produced was 4.6 lb. The off-gas treatment components for this run consisted of a condenser, electrostatic bubble scrubber, and Cambridge absolute filters. Except for a moderate burst of ruthenium that occurred during the final portion of the calcination period, the decontamination factor for ruthenium in the off-gas after it passed through the absolute filters was about 1×10^9. The ruthenium found in the electrostatic bubble scrubber liquid (which was not changed during the run) amounted to 0.06% of the original ruthenium present in the 1WW waste. Ninety-five percent of this accumulation occurred during the ruthenium burst near the end of the run. Most of the remaining ruthenium in the off-gas was retained by the absolute filters, even during the burst; and no radioactivity was detected in the hot-cell exhaust. The ruthenium found in the condensate amounted to 12% of the ruthenium initially present in the 1WW waste.

In the second run, 20 liters of 1WW waste with sodium nitrate added (1 mole of $NaNO_3$ per liter of 1WW waste) was calcined. The resulting calcine melted. The weight of the melt was 4.4 lb, and its estimated density was 2.2 g/cm^3. The melt will be used to determine the extent of radiolysis of the sodium nitrate.

In this run, only the condenser and absolute filters were used for decontamination of the off-gas. No ruthenium was detected in the cell exhaust. However, significant quantities of cesium volatilized during the meltdown step. This was the first time in the hot-cell pilot-plant program that volatilization of cesium had been observed. It is assumed that the volatilization of cesium was related to the very high sodium content of the feed and to the relatively low resultant sulfate concentration. This is in agreement with the results of tracer-level-activity experiments[10] which showed that the volatilization of cesium is suppressed by sulfate ion.

Final Disposal Methods

Engineering, Economics, and Safety Evaluation

As part of its continuing study of the economics of high-level-activity waste management,[15] ORNL has prepared a conceptual design and cost estimate for interim storage of calcined wastes in water-filled canals[16] prior to shipment of the calcined wastes to a final disposal site.

The interim storage facility[2,16] was designed to handle cylinders of calcined waste produced by a pot-calcination plant.[17] From a power-reactor fuel-processing plant, Purex waste from the recovery of 1500 tons of uranium converter fuel per year, irradiated to 10,000 Mwd/ton, and Thorex waste from the recovery of 270 tons of thorium converter fuel per year, irradiated to 20,000 Mwd/ton, were converted to solids and calcined in cylinders 6, 12, and 24 in. in diameter by 11 ft high. Calcination after storage of the wastes as both acid and neutralized solutions was considered, as was the production of a glass from acid Thorex waste. Interim solids storage periods of 1, 3, 10, and 30 years in water-filled canals were considered for cylinders which contained calcined contents that were aged for 120 days, 1 year, 3 years, and 10 years in liquid-waste storage tanks (to allow for decay of fission-product activity) prior to calcination.

A central facility canal was designed for receiving cylinders of solid waste from the calcination plant, for storing them briefly to permit the detection of defective containers, and for routing them to adjoining interim storage canals. It also served for the underwater loading of the cylinders into casks prior to shipping off-site. The central facility canal was 30 ft wide, 40 ft deep, and varied in length from 24 to 146 ft, depending on the number and size of the interim storage canals.

The depth of the canals was a function of the depth of water required to attenuate the radiation dosage to 0.25 mr/hr at the surface. The depth of the water required for shielding varied from 15.4 ft for 120-day-old acid Purex waste to 10 ft for 10-year-old reacidified Thorex waste, which, together with an additional fixed depth of 13 ft for cylinder height and freeboard, determined the total depth.

Total costs were determined for handling each type of waste separately (acid and reacidified Purex, acid and reacidified Thorex, and Thorex glass) and for the combinations of acid Purex–acid Thorex and reacidified Purex–reacidified Thorex wastes. Highest costs were for the storage of 120-day-old wastes and ranged from 19×10^{-3} mill/kw(e)-hr for 30-year storage of reacidified Thorex waste to 1.8×10^{-3} mill/kw(e)-hr for 1-year storage of acid Purex. Lowest costs were for storage of 10-year-old wastes, which ranged from 18×10^{-3} mill/kw(e)-hr for 30-year storage of reacidified Thorex to 1.5×10^{-3} mill/kw(e)-hr for 1-year storage of acid Purex. The storage of acid wastes as solids was cheaper by factors of from 2 to 2.7 than the storage of the same wastes as liquids.[18] The costs of storage of Purex and Thorex wastes together in the same facility ranged from 1.5×10^{-3} mill/kw(e)-hr for 1 year of storage to 4.8×10^{-3} mill/kw(e)-hr for 30 years of storage for the calcined acid wastes, and from 1.8×10^{-3} to 6.3×10^{-3} mill/kw(e)-hr for the calcined reacidified wastes. However, for most storage times, the storage of neutralized liquid wastes was slightly cheaper than that of reacidified Purex and Thorex calcined solids. A comparison of the costs for the interim storage of liquid and calcined wastes is shown in Fig. IV-2.

ORNL has also issued a summary report[19] on the effect of fission-product removal on the cost of high-level-activity waste management.[20]

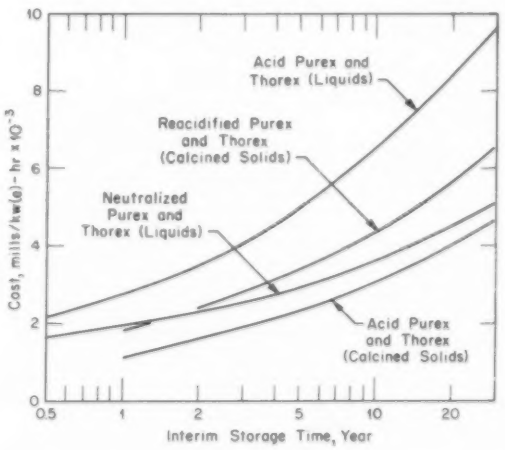

Fig. IV-2 Comparative costs of interim storage of liquid and calcined wastes.[2]

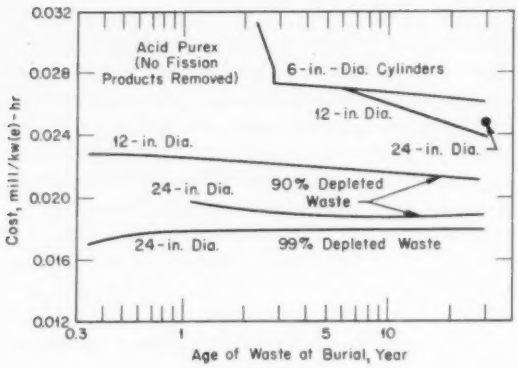

Fig. IV-3 Total costs for interim storage, calcination, shipment, and disposal in a salt mine.[19]

It was assumed for this study that, after removal (90 and 99%) of the fission products, the waste was identical to neutralized Purex waste in volume and composition of major ingredients. The sequential steps in the management of the waste from the processing of 1500 metric tons of uranium converter fuel per year irradiated to 10,000 Mwd/metric ton were: interim storage of liquid waste, conversion to solids by pot calcination, interim storage of calcined solid waste, shipment of 1000 miles, and final disposal in a salt mine.

The study indicated that fission-product removal would not significantly decrease waste management costs. The cost of managing wastes from which 99% of the fission products was removed was shown to decrease only about 30% over those of untreated wastes. The results of the study are shown in Fig. IV-3. In this figure a break is shown in the curve for untreated acidic Purex waste that is calcined in 6-in.-diameter cylinders. This break occurs because the largest shipping cask, which results in lowest costs, cannot be used prior to a waste age of 2.8 years. The total waste management cost for acid Purex was shown to decrease as the age at burial increased; and, at 30 years, the upper limit used in the study, the cost for acid Purex waste was about 0.024 mill/kw(e)-hr using both 12- and 24-in.-diameter cylinders. Costs for depleted wastes were cheaper using 24-in.-diameter cylinders and were not so strongly affected by age; these costs ranged from 0.017 to 0.019 mill/kw(e)-hr for both 90 and 99% depleted wastes for ages from 0.33 to 30 years. Relatively little cost reduction (about 7%) is achieved by increasing the fraction of fission products removed from 90 to 99%.

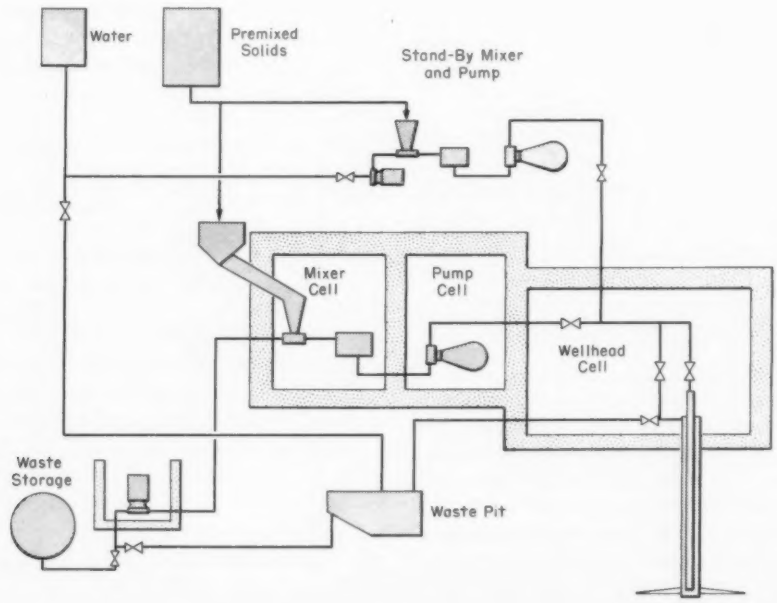

Fig. IV-4 Flow diagram of fracturing disposal pilot plant.[2]

Disposal by Hydraulic Fracturing into Shale

ORNL is continuing its investigation to determine the feasibility of disposing of liquid radioactive waste by mixing it with cement, or some other hardening agent, and then injecting the slurry deep into bedded rock through a specially constructed well.[2,21] The pressure developed at the bottom of the well forces the rock to fracture. Under favorable circumstances the fracture occurs along the bedding planes and thereby allows the waste–cement mixture to flow out into the rock in a thin horizontal sheet and then harden. This method may make possible the disposal of the large volumes of intermediate-level-activity wastes that are produced at ORNL.

ORNL is designing a waste-disposal pilot plant to handle wastes by hydraulic fracturing. A schematic flow diagram of the equipment is shown in Fig. IV-4. The waste solution will be stored on site in two 30,000-gal underground tanks. It will be pumped from the tanks to the mixer and mixed with the premixed solid constituents of the mix. The resulting mix will be pumped down the injection well by the high-pressure injection pump. The probable flow rate will be about 180 gal/min at an estimated surface pressure of 1400 psi. At the end of a run with radioactive wastes, several hundred gallons of uncontaminated cement mix will be pumped down the well to serve as a plug. This will be followed by a rubber cementing plug (to clean the cement from the tubing walls and to keep separate the cement and the water that follow it); the cementing plug will be forced down the tubing with water. The well will then be held under pressure until the cement has had time to set. Figures IV-5 and IV-6 show preliminary designs of injection wells to be used in the pilot-plant experiments. These designs were developed for ORNL on a subcontract.

Prior to the pilot-plant injection experiments with radioactive wastes, three injection experiments are planned in which water or water containing a fluid-loss additive (reduces the loss of fluid from waste slurry during hydraulic fracturing operation) will be used. The first experiment will be an injection of about 5000 gal of water into the injection well to determine the fluid-retention properties of the fracture. The second will be an injection of about 50,000 gal of water into the same fracture to determine the effect of injection volume on the fluid-retention properties. The third experiment will be an injection of about 50,000 gal of water and fluid-loss additive into a new fracture to determine the effect of the additive.

Protection for Buried Solid Wastes

The Savannah River Plant is field-testing Wyoming bentonite as a cover material for preventing the leaching of buried solid wastes, such as contaminated equipment.[22] This type of waste has accounted for more than 99% of the radioactivity committed to the ground at the

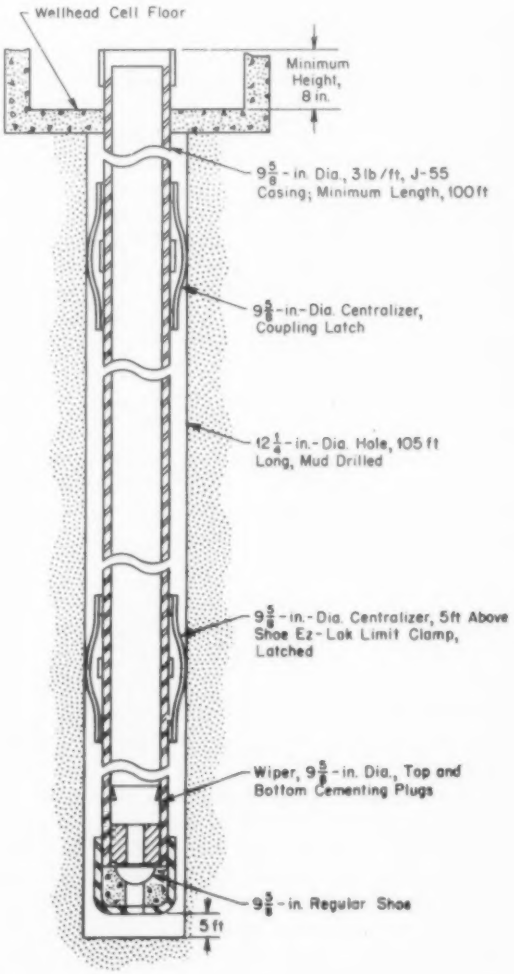
Wellhead Cell Floor
Minimum Height, 8 in.
9⅝-in. Dia., 31 lb/ft, J-55 Casing; Minimum Length, 100 ft
9⅝-in-Dia. Centralizer, Coupling Latch
12¼-in.-Dia. Hole, 105 ft Long, Mud Drilled
9⅝-in.-Dia. Centralizer, 5 ft Above Shoe Ez-Lok Limit Clamp, Latched
Wiper, 9⅝-in. Dia., Top and Bottom Cementing Plugs
9⅝-in. Regular Shoe
5 ft

Fig. IV-5 Surface string of the Oak Ridge waste-disposal well.[2]

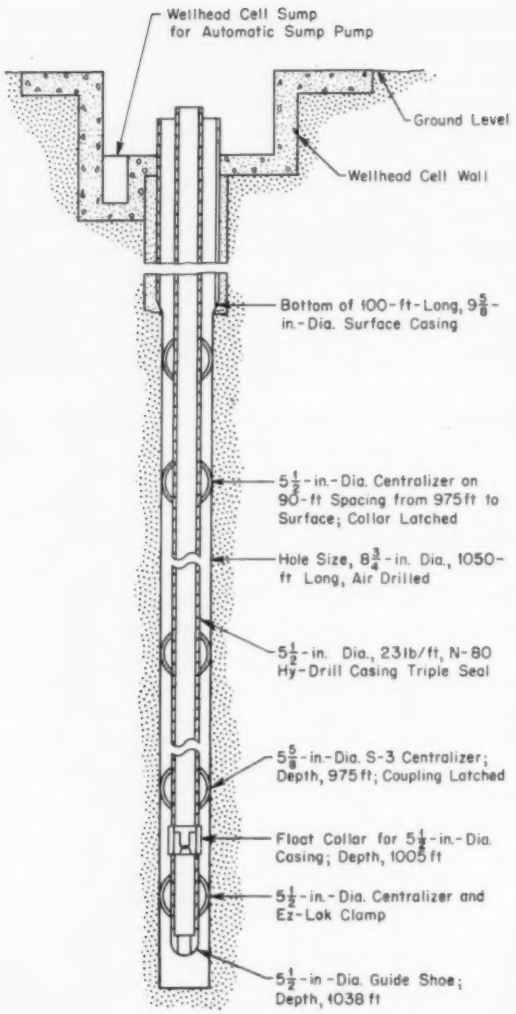
Wellhead Cell Sump for Automatic Sump Pump
Ground Level
Wellhead Cell Wall
Bottom of 100-ft-Long, 9⅝-in.-Dia. Surface Casing
5½-in.-Dia. Centralizer on 90-ft Spacing from 975 ft to Surface; Collar Latched
Hole Size, 8¾-in. Dia., 1050-ft Long, Air Drilled
5½-in. Dia., 23 lb/ft, N-80 Hy-Drill Casing Triple Seal
5⅝-in.-Dia. S-3 Centralizer; Depth, 975 ft; Coupling Latched
Float Collar for 5½-in.-Dia. Casing; Depth, 1005 ft
5½-in.-Dia. Centralizer and Ez-Lok Clamp
5½-in.-Dia. Guide Shoe; Depth, 1038 ft

Fig. IV-6 Long string of the Oak Ridge waste-disposal well.[2]

Savannah River Plant. Although the annual local rainfall is about 45 in., the radionuclides from this waste have not, apparently, migrated into the groundwater. Nevertheless, leaching of the radionuclides may ultimately become a problem.

Since all common construction materials such as concrete and asphalt are subject to physical and chemical deterioration, long-lasting natural materials were investigated. Laboratory studies with kaolinite, Wyoming bentonite, native soil, and various mixtures of the three indicated that a layer of bentonite covered with soil would be best.

The effectiveness of a bentonite layer in soil exposed to natural conditions was initially determined in lysimeters under field conditions. Combinations of 1- and 2-in. layers of bentonite covered with 1- and 2-ft layers of soil were compared with untreated soil. During 16 months of testing in which there was 75 in. of rain, 28 in. of the rain had passed through the untreated soil but none had passed through the bentonite layers. Observations during a prolonged drought indicate that 2 ft of soil over the bentonite is needed to prevent the bentonite from cracking.

A large-scale field test site has been completed in which a 50- by 50-ft area, simulating an old burial zone, was ditched on all four sides to a depth of 25 ft. Fine gravel was spread 5 ft deep in the ditch to provide drainage to an adjacent ditch so the water table would not rise into the simulated burial zone. Wet bentonite (50% bentonite, 50% water) was then sprayed from a "cement gun" on the vertical inner wall of the ditch above the gravel and over the horizontal surface of the burial zone. The bentonite formed a continuous, umbrellalike layer, 2 in. thick. The ditches surrounding the treated zone were backfilled, and the zone was covered with 2 ft of soil. The bentonite covering will be evaluated by comparing the migration rates of radionuclides buried inside and outside of the treated zone and by measuring the soil moisture.

Recovery of Fission Products

As part of the Waste Management Program at Hanford, solvent-extraction processes for the extraction of long-lived heat-generating fission products from various Purex-plant wastes are being developed.[23] A new solvent-extraction process (CSREX) is being developed[6,24] which makes possible the simultaneous extraction of cesium, strontium, and rare earths from Purex formaldehyde-treated process waste by means of a single solvent at a single *p*H. It is based on the synergistic effect of combining two solvents, di-2-ethylhexyl phosphoric acid (D2EHPA) and 4-*sec*-butyl-2-(α-methylbenzyl) phenol (BAMBP). D2EHPA was used at Hanford for the multicurie-scale recovery of strontium.[25] It will also coextract rare earths (but not cesium) from weakly acidic feed solutions.

High-molecular-weight phenols,[26] which were initially investigated at ORNL, were found to extract cesium (but not strontium) from highly alkaline solutions. [In the ORNL study, phenols very effectively removed (about 99%) cesium from simulated Purex 1WW wastes adjusted to *p*H 12.6.] In the CSREX flow sheet (Fig. IV-7), a mixture of D2EHPA and BAMBP in a kerosene type diluent coextracts cesium, strontium, and rare earths at a *p*H of about 4 to 6 (Fig. IV-8). The cesium-strontium and rare-earth fractions are then separated from each other by selective stripping. The recovered strontium is readily separated from the cesium by means of a carbonate precipitation.

A solvent-extraction method that employs a solution of dipicrylamine dissolved in nitrobenzene[27] for the removal of cesium from Hanford process wastes is described in two topical reports.[28,29] Bray and Roberts[28] have summarized the laboratory and hot-cell investigations of this process study. Richardson[29] has described the semiworks-scale demonstration runs (with simulated Purex-process wastes) that were carried out in 3-in.-diameter by 9-ft-high pulse columns.

Ion-Exchange–Solvent-Extraction Recovery of Cesium at ORNL

ORNL has demonstrated in preliminary tests the effective recovery of cesium from simulated Purex-process waste (1WW) by a process that involves the adsorption of the cesium on ammonium molybdophosphate (AMP), dissolution of the AMP in caustic, and extraction of the cesium from the resulting solution with 4-*sec*-butyl-2-(α-methylbenzyl) phenol (BAMBP).[30] In an experiment with simulated Purex 1WW that contained 0.37 g of cesium per liter and added Cs^{134} tracer, the loaded AMP was dissolved in NaOH, resulting in a solution with a *p*H of 11.7. This solution was adjusted to a *p*H of 13.2. The concentration of cesium in this solution was 5.4 g/liter. It was then contacted with five successive 0.2 volumes of 1*M* BAMBP in diisopropylbenzene (DIPB). This extraction step reduced the cesium concentration in the aqueous phase to 0.009 g/liter, corresponding to a cesium recovery of >99.8%. Extraction coefficients for cesium ranged from 4 to 26 for the extraction step. The first two organic fractions were combined and scrubbed with two successive 0.2 volumes of 0.05*N* NaOH. The extraction coefficient for cesium was about 3 for

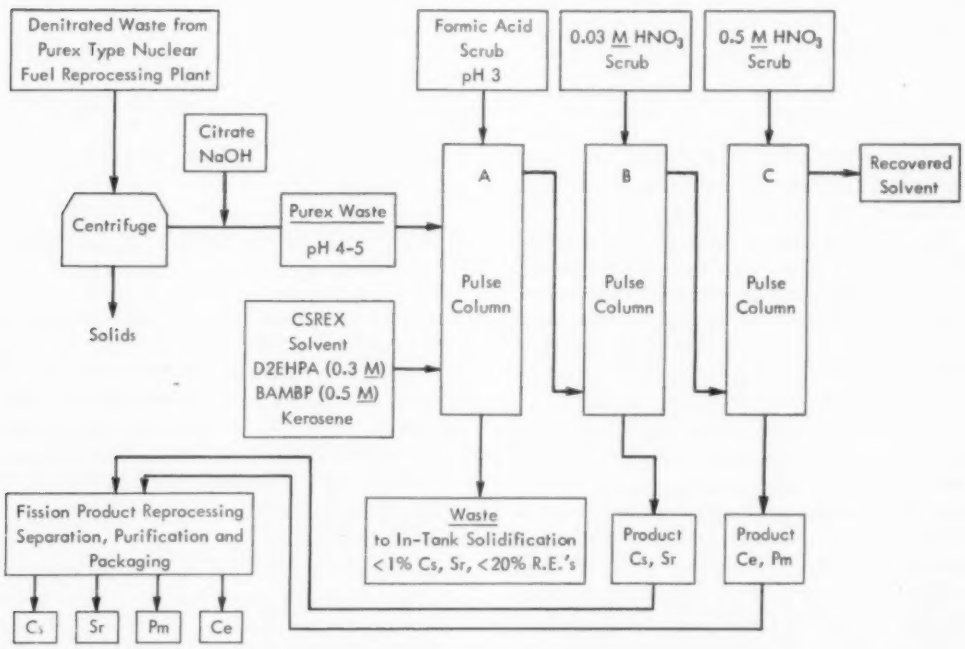

Fig. IV-7 CSREX process flow sheet.[6]

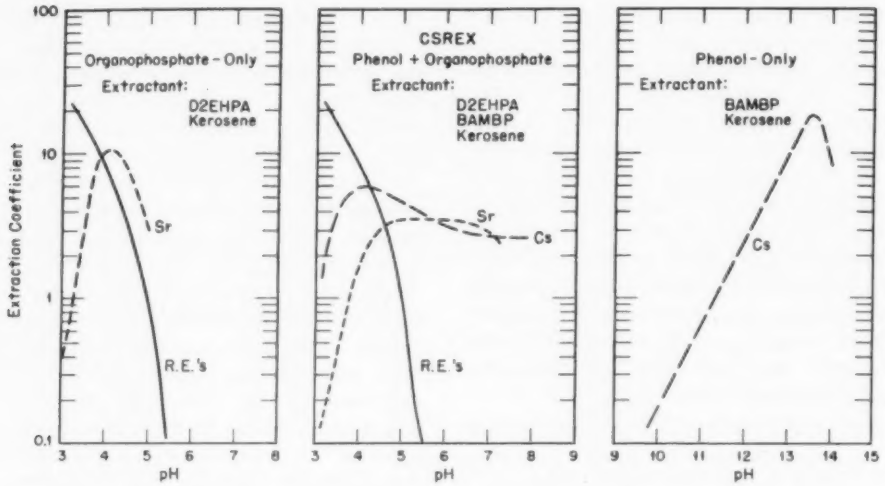

Fig. IV-8 Coextraction of strontium, cesium, and rare earths.[6]

the scrubbing step. The scrubbed extract was stripped quantitatively by a single contact with 0.33 volume of 0.3*N* HNO_3, which resulted in a product solution containing 28.3 g of cesium per liter. The separation from contaminants was highly effective; the cesium product solution contained only 0.021 g of sodium per liter, 0.06 g of ammonia per liter, < 0.01 g of molybdenum per liter, and <0.003 g of phosphate per liter.

Ion-Exchange Recovery of Cesium at Harwell

Harwell has issued a report[31] that describes a proposed ion-exchange process for the recovery of Cs^{137} from fission-product waste concentrates (Butex-process wastes). The process utilizes two inorganic ion exchangers, ammonium 12-molybdophosphate (AMP) and zirconium phosphate (ZP). The cesium is preferentially adsorbed on columns of coarse particles of AMP, followed by a washing step with dilute nitric acid which quantitatively removes all other activities except some Zr^{95}-Nb^{95}. Recovery of the cesium is achieved by dissolving the cesium-loaded AMP column in sodium hydroxide. The resulting alkaline solution is filtered to remove a small amount of residual precipitate and then adjusted to *p*H 4. This neutralized solution is then passed through the ZP column, which adsorbs the cesium and effects a separation of cesium from sodium. Cesium is finally recovered from the ZP column by elution with hydrochloric acid. The cesium chloride produced has a high chemical and radiochemical purity.

The principal advantages of this proposed method over other methods such as recrystallization and precipitation are stated to be:

1. Small size of equipment, leading to a great reduction in shielding requirements, and simplicity of operation
2. Adaptability to virtually any waste composition or concentration
3. No nonvolatile, and little volatile, materials are added to the waste
4. Prior neutralization of the waste is not necessary

AEC Symposium on Packaging and Regulatory Standards for Shipping Radioactive Materials

The proceedings[32] of an AEC symposium on packaging and regulatory standards for shipping radioactive materials have become available. The meeting was held in Germantown, Md., on December 3–5, 1962. Papers were presented which discussed the problems of transportation of radioactive materials, the regulatory standards for shipping-container design, and the testing programs related to containers for transportation of radioactive materials.

Corrosion in Waste Processing

ORNL is continuing its efforts to reduce the volume of aqueous wastes through a pot-calcination process. This process is basically a two-

step process in which aqueous wastes are continuously evaporated until sufficient solids have been accumulated, after which the solids are calcined by heating at temperatures up to 1050°C.

A type 304L stainless-steel calcination pot was subjected to an evaporation-calcination cycle using synthetic Hanford Purex wastes while loaded to 315 psi. The physical stability of the stainless steel is of considerable interest, in addition to its corrosion-resistance characteristics. After heating of the pot to 1050°C for 96 hr, one side of the pot showed an elongation of 1.49×10^{-3} in./in., whereas the other side showed no measurable change. This indicates that the total elongation of an 8-ft-long pot during a normal evaporation-calcination cycle (estimated at less than 48 hr) should be less than 0.1 in.

During these tests, welded specimens of type 304L stainless steel, titanium-45A, and Hastelloy F were exposed in the Purex waste solution, at the interface, and in the vapor. Weight gains during this exposure varied from 0.2 mg/cm^2 for Hastelloy F exposed for 552 hr in the vapor, to 5 mg/cm^2 for titanium-45A exposed for 840 hr in the solution. After defilming was completed, the stainless-steel type 304L specimens were observed to have corroded at rates of 0.41, 0.34, and 0.14 mil/month in 192 hr of exposure in the vapor, at the interface, and in the solution, respectively.[33] An additional test was performed in this system to determine whether austenitic stainless steels have different corrosion rates in superheated vapors than in condensing vapors, as might be expected. The temperature of the superheated vapor varied between 150 and 305°C. In 1008 hr of exposure, the corrosion of stainless-steel types 347 and 304L which had been exposed to the superheated vapors was less than 0.1 mil/month. The corrosion rate for exposure condensing vapors was 2.6 to 3.3 mils/month. It appears that a stainless-steel head assembly for the fixation pot will have excellent durability if its temperature can be maintained above 150°C.

At Hanford, a radiant-heat spray-calciner column is being studied as a means of reducing the volume of aqueous wastes. Following a total of 11 runs with various types of aqueous wastes, the calciner column, which is constructed of 16-gauge Inconel, was thoroughly washed down and descaled. In the process a leak was discovered in the column. Full vacuum could not be drawn; the size of this hole was estimated (from pressure-drop measurements) to be about 0.025 sq in. The leak was thought to be caused by a cracked or corroded weld, or perhaps to general corrosion of the shell surface. Detailed information regarding exposure conditions was not included in the report. It is now thought that the most promising alloys for this particular environment are a 50-50 chromium-nickel alloy or a 50-25-25 chromium-nickel-iron alloy. Corrosion tests of Inconel and other alloys are now under way.[6]

At Brookhaven National Laboratory a study is being made of the ultimate disposal of high-level wastes, with major emphasis being placed on the formation of phosphate glasses as a means of incorporating the fission products into stable media. Of primary interest is the development of a process in which the entire conversion from the raw aqueous wastes to the final glass product would be carried out in an all-liquid system. A platinum crucible was used in the study of the continuous production of phosphate glass. The crucible has been subjected to a variety of conditions for a number of months on an intermittent basis, usually for 6- to 8-hr periods. It is estimated that the period of exposure of the platinum crucible to molten glass may be considered equivalent to full-scale plant operation for some 500 hr with Purex feed. No evidence of corrosion of the platinum crucible has been found.[34]

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Section

V

Reactor Fuel Processing

Production of Uranium, Thorium, Plutonium, and Their Compounds

Production of Uranium Dioxide

Fluid-Bed Reduction of Uranium Trioxide

Gas-solid reactions, particularly the reduction of uranium trioxide with hydrogen and the hydrofluorination of uranium dioxide to produce uranium tetrafluoride, are important steps in the production of both uranium metal and uranium hexafluoride. The rate of these reactions has been shown to be determined by the structure of the solid surface which, in turn, is modified during the progress of the reactions. Since thermal damage of the solid surface is significant in this type of heterogeneous reaction, considerable attention has been directed toward employing fluid-bed reactors in the preparation of uranium dioxide and tetrafluoride. Fluid-bed reactors offer the advantage of efficient gas-solid contact and temperature control.

An integrated fluid-bed system[1] has been developed at the Weldon Spring Plant of the Mallinckrodt Chemical Works for the production of high-quality UF_4 from uranyl nitrate solution. The reaction sequence involved denitration of uranyl nitrate liquors to uranium trioxide, followed by the hydrogen reduction of UO_3 to UO_2, and, finally, hydrofluorination of UO_2 to produce UF_4.

Demonstration tests have indicated that sustained operation of the continuous fluid-bed denitrator at rates greater than 350 lb UO_3/(hr)(sq ft of reactor cross section) is practical with uranyl nitrate feed concentrations up to 11.8 lb of uranium per gallon. It was observed that the physical characteristics of the UO_3 product can be varied by minor changes in the operating conditions. Experimental results indicated that an increase in bed temperature, feed concentration, sulfate content in the feed stream, or production rate will generally result in an increase in the mean particle size of the UO_3 product.

High-quality UO_2 has been consistently produced by the reduction of fluid-bed UO_3 with dissociated ammonia in a tapered single-stage fluid-bed reactor. The reactor was designed with an internal tapered mandrel which permits a close approach to piston flow of the solids in the fluid-bed reactor, provided that the gas velocity does not greatly exceed that required for incipient fluidization. Although it was not possible to operate the reactor under piston-flow conditions, high-quality UO_2 was produced in the 5-in.-diameter single-stage pilot-plant reactor at a rate of 100 lb UO_2/(hr)(cu ft of reactor volume). Results of demonstration tests showed that increased bed temperature, increased hydrogen excess, and decreased production rate all increased the amount of conversion of UO_3 to UO_2. A significant result of the tests was that the amount converted to UO_2 increased as gas flow increased in spite of the additional solids mixing incurred.

Hydrofluorination of uranium dioxide to uranium tetrafluoride is accomplished by a two-stage reactor system. Initial tests showed that UF_4 of acceptable purity could not be produced at satisfactory rates in a single-stage reactor. Low hydrogen fluoride consumption was realized through the use of a low excess of HF in the second-stage reactor and by the recycle of HF to the first stage as the HF-H_2O azeotrope.

Reduction of Hydrated UO_3

Dell and Wheeler[2] at Harwell have observed that, when UO_3 produced by spray denitration of uranyl nitrate in a fluidized-bed reactor is reduced by hydrogen, a relatively unreactive grade of UO_2 is the product. A series of experi-

mental tests were performed to evaluate an activation procedure based on the conversion of uranium trioxide to a hydrated species followed by dehydration and reduction. The results of these tests, reported in the October 1962 issue of *Reactor Fuel Processing*,[3] showed that the reactivity of UO_3 toward reduction to UO_2 and subsequent hydrofluorination to UF_4 depends on the conditions of forming the hydrated uranium trioxide and on the conditions of dehydration. The production of a reactive grade of uranium dioxide is favored by (1) hydration of uranium trioxide under conditions that minimize crystal growth, (2) hydration to the dihydrate rather than the monohydrate, and (3) decomposition of the hydrate and reduction to uranium dioxide at 400°C.

Reduction of Uranyl Formate

Methods are being evaluated at Savannah River[4] for the preparation of uranium oxide from uranyl nitrate solutions. One method that appears attractive for the conversion of uranium to either UO_2 or U_3O_8 involves the conversion of uranyl nitrate to uranyl formate by ion exchange and the conversion of uranyl formate to UO_2 in a fluidized bed. Uranyl formate decomposes at 400°C to produce U_3O_8 in air or UO_2 in an inert gas such as argon.

A study of the thermal decomposition of uranyl formate was carried out in laboratory tests. The results indicate that uranyl formate decomposes in several steps: (1) water of hydration is evolved below 160°C, (2) CO and CO_2 are evolved and an intermediate product ($UO_2 \cdot H_2O$) is formed at 300°C, and (3) anhydrous UO_2 is formed at about 450°C. The decomposition sequence may be represented as

$$UO_2(HCOO)_2 \cdot H_2O \xrightarrow{110-160^\circ C} UO_2(HCOO)_2 + H_2O \quad (1)$$

$$UO_2(HCOO)_2 \xrightarrow{300^\circ C} UO_2 \cdot H_2O + CO_2 + CO \quad (2)$$

$$UO_2 \cdot H_2O \xrightarrow{450^\circ C} UO_2 + H_2O \quad (3)$$

There appears to be evidence that the second step occurs in two stages: $UO_2(HCOO)_2 \rightarrow UO_3 \cdot H_2O + 2CO$ and $UO_3 \cdot H_2O + CO \rightarrow UO_2 \cdot H_2O + CO_2$. Therefore the production of UO_2 would depend strongly on the conditions of preparation since one-half of the CO evolved in the first stage must be used to reduce the hydrated uranium trioxide in the second stage. Results of tests employing shallow fluidized beds showed that the yield of UO_2, under otherwise identical conditions, was reduced by increasing the flow of inert gas but was increased to 100% when CO was added to the gas stream. It is expected that excess ammonium formate and formic acid in the uranyl formate feed stream would provide, on decomposition, sufficient CO in the fluid bed to reduce the uranium to uranium dioxide.

Coating of Uranium Dioxide Particles

The encapsulation of individual uranium dioxide particles with ceramic coatings by fluidized-bed techniques has been demonstrated by workers at Battelle Memorial Institute (BMI).[5] A description of the procedures used in coating particles with alumina by hydrolysis of aluminum chloride vapor appeared in a previous Review.[6] This technique has been extended to include the deposition of beryllium oxide and magnesium oxide.[5]

Alumina coatings deposited at temperatures from 500 to 700°C are porous and of low density, and X-ray diffraction examination indicates that they are completely amorphous. However, the coatings become crystalline on heating at temperatures of about 1000°C and above. Alumina coatings of near theoretical density are deposited at 1000°C. These coatings are impermeable and consist of small crystallites of alpha alumina dispersed in an amorphous matrix. Coatings deposited at 1400°C are highly crystalline but exhibit a coating integrity inferior to that of coatings produced at 1000°C (Ref. 5).

Beryllia coatings, prepared by hydrolysis of $BeCl_2$ vapor, require a higher deposition temperature than alumina coatings in order to achieve equivalent properties. Coatings deposited at 800 to 1100°C are not crystalline and are porous. Beryllia coatings prepared at 1400°C are of near theoretical densities, are crystalline, and have good integrity.

Uranium dioxide particles may be coated with a layer of magnesium oxide by hydrolysis of magnesium iodide vapor in a fluidized bed. Transparent, dense, and void-free coatings are obtained at 1400°C. Magnesia coatings on a dense UO_2 substrate tend to crack when the particles are cooled from the temperature of coating. This is apparently due to the large difference in the thermal-expansion coefficients of uranium dioxide and magnesia.

In order to minimize the effects of irradiation and temperature on the coated particles, certain modifications in the coating procedure

have been investigated.[5] These modifications include incorporation of interlayers of materials to absorb and retain fission recoils without damaging the outer coating, and the use of void regions within the coating or fuel to permit dimensional changes, promote reduction in fission-gas pressure, or serve as barriers to crack propagation. An example of this type of structure is pyrolytic carbon coating over an inner layer of Al_2O_3. The purpose of the Al_2O_3 is to protect the pyrolytic carbon from direct fission-recoil damage and also to provide a material in which fission products will lodge and through which they will diffuse slowly.

Uranium dioxide particles may be coated with pyrolytic carbon at temperatures of from 950 to 2000°C by the decomposition of acetylene or methane in a fluidized-bed reactor.[5] The structure of 1400°C pyrolytic coating varies from columnar to laminar depending on the deposition rate. The columnar type of coating appears as radially oriented grains, whereas the laminar coating consists of concentric spherical shells. Coatings applied at rates of less than 10 μ/hr are columnar in appearance, whereas those applied at higher rates have a laminar appearance.

Davies and associates[7] have investigated the vapor deposition of zirconium carbide on uranium dioxide particles. Zirconium carbide coatings were produced by the reaction between zirconium tetrachloride vapor and methane with hydrogen as a carrier gas. A deposition temperature of 1500°C was required for the production of dense adherent coatings. Process variables were studied in both graphite fluid-bed equipment and in a graphite rotating-crucible apparatus. In general, more satisfactory coatings were obtained in the rotating crucible, since agglomeration of particles occurred frequently in fluid-bed coating tests.

Preparation of U(IV) Nitrate Solutions

Considerable interest has been shown in the use of U(IV) solutions as reducing agents in the solvent-extraction separation and recovery of plutonium from uranium. The reducing agent now employed, ferrous sulfamate, would be replaced by U(IV) nitrate. Since U(IV) nitrate adds no extraneous ion to the process, both the corrosion rate of the waste evaporators and the amount of radioactive waste to be stored would be decreased.

A prototype electrolytic converter for the production of U(IV) nitrate from uranyl nitrate has been developed at Savannah River.[4] Laboratory tests have indicated that a batch cell with a metal (niobium) cathode is as effective as a flow cell designed with a porous graphite cathode. At the niobium cathode the electrochemical reduction of U(VI) to U(IV) competes with the production of cathodic gases (H_2 or nitrogen oxides). Oxygen is produced at the platinum anode, but any U(IV) that migrates to the anode is reoxidized to U(VI) with a resulting loss of yield. An inert ceramic diaphragm, installed between the electrodes, reduces the reoxidation at the anode but increases the competition between the production of U(IV) and the cathodic gases. It was observed that an increase in current density increased the consumption of current per mole of U(IV) produced. This effect became very pronounced as the concentration of U(VI) decreased below 0.1*M*.

Carroll, Burns, and Warren at Hanford Atomic Products Operation (HAPO)[8] have investigated the photoactivated reduction of uranyl nitrate to prepare U(IV) in a nitrate solution. Ultraviolet radiant energy of wavelength below 3500 A is effective in activating the reduction of uranyl solutions. Greater reduction rates were obtained with formaldehyde as the reducing agent than with hydrazine, methanol, formic acid, sulfamic acid, or urea. Initial net reduction rates were found to exhibit a 0.30-power dependence on formaldehyde concentration in the range of 0.05*M* to 0.2*M*. In tests employing higher concentrations of formaldehyde, the rate dependence on formaldehyde concentration was slightly greater. The effect of temperature on reduction varied considerably, depending on the reducing agents employed. Although increased temperature would be expected to increase the rate of reduction of photoactivated uranyl ions, the presence of certain reducing agents causes the rate of deleterious side reactions to increase more rapidly with a resulting decrease in U(IV) production. Raising the temperature from 30 to 70°C increased the rate of reduction in hydrazine systems, but, with the use of formaldehyde, an increase in temperature resulted in a substantial decrease in U(IV) formation.

The results of these tests indicated that the photoactivated reduction process appears applicable on either a batch or a continuous basis.

Calculations have indicated that the cost of producing U(IV) from uranyl nitrate is less than 10 cents per pound, with nearly the entire cost being assessed to the lamp.

Production of Plutonium Metal

Electrodeposition of Plutonium

Brodsky and Carleson[9] at Argonne National Laboratory (ANL) have investigated the electrodeposition of plutonium from molten-salt solutions of Cs_2PuCl_6. The electrolyte employed in the process was a 5 mole % solution of Cs_2PuCl_6 in a LiCl-KCl eutectic. A $\frac{1}{8}$-in.-diameter graphite rod was used as the anode, and a $\frac{1}{8}$-in.-diameter tantalum rod was used as the cathode. Tests were carried out in Vycor electrolysis cells at temperatures between 685 and 715°C. Molten plutonium metal was deposited at the tantalum cathode.

Up to 66% of the plutonium dissolved in the salt was recovered as a deposit in the tests. When molten plutonium metal was used as the cathode, no net plutonium was recovered because of reoxidation of the metal by chlorine dissolved in the electrolyte. It was observed during the investigation that the efficiency of the electrodeposition process was severely limited by reactions between the chlorine generated at the anode and the deposited metal. This limitation requires that the reverse reaction must be significantly reduced before the process can have widespread application. Chlorination of the plutonium metal product might be prevented by (1) constructing a cell with separate anolyte or catholyte compartments, (2) flushing the cell with an inert gas to remove rapidly the chlorine produced at the anode, or (3) removing the metal deposit frequently.

Plutonium trichloride, used in the preparation of Cs_2PuCl_6, is very hygroscopic and, therefore, difficult to store. Workers at ANL[10] have investigated a method of preparing plutonium trichloride directly in a fused-salt medium. The procedure involves the chlorination of plutonium metal by tantalum pentachloride in a KCl-LiCl eutectic. From this molten salt, pure plutonium metal may be recovered by electrolysis using tantalum cathodes.

Mullins and associates at Los Alamos Scientific Laboratory (LASL)[11] have recently developed a process for producing plutonium metal of high purity by an electrorefining procedure. A detailed description of the plutonium electrorefining process was presented in a previous Review.[12] Briefly, the process involves the oxidation of impure plutonium metal at the anode to form trivalent plutonium ions in the molten-salt electrolyte, which is maintained at a temperature above the melting point of plutonium metal. The plutonium ions are transported through the electrolyte to a tungsten cathode, where they are reduced to metal. The molten-metal product drains down the cathode into a product reservoir. The process has been demonstrated on a 3.5-kg scale and has direct application in the production of high-purity metal and the recovery of plutonium from metal scrap.

Bomb Reduction of Plutonium Chloride and Plutonium Fluoride

The main objection to the use of plutonium fluoride in plutonium metal preparation is the high neutron density resulting from intense alpha radiation of the fluorine atoms. The use of a halogen of higher atomic number would cause the neutron exposure to be reduced more than by any reasonable shielding. Therefore a process was developed at HAPO[13] for the preparation of plutonium metal by the bomb reduction of plutonium trichloride with calcium.

Plutonium trichloride is reduced by calcium in the presence of an iodine booster. Process conditions for the bomb-reduction step were established in a series of tests[13] employing reduction charges ranging from 30 to 700 g. The plutonium trichloride used in these tests was prepared by chlorination of plutonium dioxide by phosgene (a description of the process was presented in a previous Review[14]). The optimum amounts of calcium metal and iodine, as determined by the yield of plutonium metal, ranged from 15 to 30% excess calcium and 0.2 to 0.3 mole of iodine per mole of plutonium. A major problem associated with the reduction process was the difficulty of obtaining a satisfactory liner material for the bomb-reduction vessel. The molten calcium chloride slag formed during the reduction penetrated the liner material, and, upon cooling, the charge and liner became cemented inside the pressure vessel. Of several materials tested, a crucible liner fabricated from magnesium oxide appeared most satisfactory.

The presence of moisture in the plutonium trichloride up to 0.6 wt.% would not affect the reduction operation. Satisfactory reductions were obtained with plutonium trichloride feed material having chlorine-to-plutonium mole ratios of from 1.7 to 3.0. Good mixing of the reduction charge prior to introduction into the bomb was not necessary; high metal yields were achieved if the plutonium trichloride was placed on top of the metal and iodine during charging of the bomb.

Savannah River[15] recently began using a process for the production of plutonium metal based on the precipitation and reduction of plutonium trifluoride. Plutonium trifluoride can be precipitated from a Pu(III) nitrate solution, dried, and reduced to metal using calcium with an iodine booster. This system, with variations, has been in routine use at the Savannah River Plant to produce 2000-g buttons of plutonium metal. The low heat of reduction of the trifluoride requires 0.6 mole of iodine per mole of plutonium as a booster for good metal yields. The problems associated with the use of an iodine booster have been eliminated by oxidizing anhydrous PuF_3 with dry oxygen to a mixture of PuF_4 and PuO_2 which has sufficient heat of reduction to be reduced with calcium alone in 96% yield. A complete description of the Savannah River plutonium operation has appeared in a previous Review.[16]

Design of Plutonium Facilities

The toxicity, susceptiveness to oxidation, radioactivity, and low criticality limits necessitate certain restrictions in the processing of plutonium compounds. The rules for plutonium foundry practices at LASL have been established[17] as:

1. All plutonium foundry operations are performed in completely enclosed glove boxes.
2. The atmosphere in the glove boxes is dried air (−10°C dew point maximum).
3. All plutonium casting and/or alloy work is performed in vacuum furnaces except when an inert atmosphere is desirable for a specific alloy.
4. Almost all heating and melting are done by induction.
5. A maximum of 6 kg of pure plutonium is permitted in any one glove-box section (a glove box is about 60 in. high by 30 in. wide by 30 in. deep). Larger limits for dilute plutonium alloys are permissible if approved by the criticality board at LASL.
6. No cooling water is used inside the casting furnaces because of the criticality problem; however, cooling with a low-hydrogen-content fluid has been approved.

Descriptions of several plutonium facilities for studying the synthesis of plutonium compounds have been published in the Proceedings of the 10th Conference on Hot Laboratories and Equipment.[18]

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